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# SOIL SCIENCE

*Founded 1916 by Jacob G. Lipman*

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# SOIL SCIENCE

FOUNDED BY
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# SOIL GENESIS FROM ANDESITE IN GRENADA, BRITISH WEST INDIES

F. HARDY AND G. RODRIGUES[1]

*Imperial College of Tropical Agriculture, Trinidad, B.W.I.*

Received for publication May 18, 1939

The island of Grenada in the British West Indies offers excellent material for the study of soil genesis from basic igneous rocks under a humid tropical climate having alternate wet and dry seasons. The commonest rocks comprise Pleistocene andesitic and basaltic lava-flows, the profound weathering of which has given rise to thick masses of red earth, from which fertile soils, mainly planted in cacao, have evolved. A typical example of a red earth profile has been examined by the writers in continuation of investigations begun in British Guiana (7).

## DESCRIPTION OF THE PROFILE

The site of the profile is a new road cutting through a descending spur near the 12th milepost on the St. Georges-Grenville Road in St. David's Parish, Grenada, at an elevation of 750 feet, where the annual rainfall is about 70 inches.

(i) The *parent rock* is a porphyritic augite-andesite, in which augite occurs as large, black, lozenge-shaped phenocrysts (15.5 per cent), set in a fine-grained, gray, feldspathic matrix consisting of labradorite (73 per cent), with accessory magnetite (7.5 per cent) and yellow olivine (4 per cent). The rock shows columnar and spheroidal jointing, and its upper parts have disintegrated into rounded boulders, the average diameter of which is 2 feet and which have split into concentric shells.

(ii) The outermost of these shells are covered with friable crusts of *primary laterite*, 1 to 3 cm. thick, colored golden-yellow, and studded with bright red-brown powdery patches, presumably comprising hydrous iron oxide residues derived from the augite phenocrysts.

(iii) Passing outward from the laterite-encrusted boulders, is a heterogeneous coherent mass, 1 to 4 feet thick, of *pink earth*, consisting of finely speckled groundmass colored pale red, blue-gray, and gray-white, in which altered augite crystals are still conspicuous as rust-brown spots. Certain areas of this

[1] The authors wish to acknowledge their indebtedness to G. Milne, of the East African Agricultural Research Station, Amani, Tanganyika, for collecting the materials of the red earth profile during a visit to Grenada in April, 1938, and for many helpful discussions; also to C. E. Marshall, of the Missouri State Agricultural College, Columbia, for the gift of mineral specimens and for valuable advice.

zone show brighter intermerging patches, colored russet, red-brown, orange, and yellow, presenting a kaleidoscopic aspect.

(iv) Upward, the pink earth passes into a layer, 2 to 8 feet thick, of homogeneous brick-red to pale reddish brown clay loam (*red earth*) showing a rough columnar structure with a brittle, coarsely granular substructure.

(v) The red earth grades into a uniform dark red-brown loamy surface soil, 1 to 5 feet thick, showing a well-developed crumb, consisting of stable angular fragments. The soil is covered with a thin leaf litter, and supports dense secondary bush.

Representative samples of the different parts of the profile were procured in the middle of the dry season of 1938 for laboratory examination and labelled (i) parent rock, (ii) laterite, (iii) pink earth, (iv) red earth, and (v) soil.

## EXPERIMENTAL PROCEDURES

The samples were examined by chemical, physical, and mineralogical methods, and by the application of dye tests, including the alizarin test elaborated by us in 1931 (5). The chief aim was the complete identification of the specific mineral components, both primary and secondary, of each of the five kinds of material.

Subsamples were ground to pass a 100-mesh sieve, and colloidal clay fractions, upper size limit 2 $\mu$, were separated by dispersion in sodium carbonate solution with mechanical agitation, followed by appropriate settling in beakers. The clay suspensions were evaporated, treated with acetic acid, filtered, washed, oven-dried, and weighed. The sand and the silt fractions were also dried and weighed. For mineralogical analysis, subsamples were merely crushed, dispersed, and fractionated by a sieve and beaker method into three sand grades (minimum size 10 $\mu$), a silt grade, and a fine silt and clay grade (maximum size 5 $\mu$). The sand grades were cleaned with boiling hydrochloric acid for examination under the microscope.

### *Chemical analysis*

Both the whole materials and the colloidal clay fractions were quantitatively analyzed after sodium carbonate fusion by standard methods of silicate analysis. Quartz was determined by the tri-acid method (7). Magnetite was removed from the whole material prior to fusion and was determined separately. Gibbsitic alumina was estimated, after ignition, by the alizarin method. The analytical data obtained are presented in table 1, and losses and gains are expressed in table 2, on the assumption that total alumina remains constant during the transformations. Table 3 expresses the results in terms of component minerals, assuming kaolinite to be the chief secondary mineral generated.

### *Mineralogical analysis*

The magnetite content of the five grades of each of the materials was determined by abstracting the mineral first with a small bar magnet, and then with

TABLE 1

*Bulk chemical composition of red earth profile samples*

| SAMPLE | PERCENTAGE COMPOSITION | | | | | | | | | | | | MOLECULAR RATIOS* | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $SiO_2$ | | $Al_2O_3$ | | $Fe_2O_3$ | | $TiO_2$ | CaO | MgO | $K_2O + Na_2O$ | $H_2O$ | | $\frac{SiO_2}{Al_2O_3}$ | $\frac{Fe_2O_3}{Al_2O_3}$ | $\frac{SiO_2}{H_2O}$ | $\frac{Al_2O_3}{H_2O}$ |
| | Quartz | Combined | Gibbsitic | Combined | Magnetite† | Other | | | | | Gibbsitic | Other | | | | |
| *Whole material* | | | | | | | | | | | | | | | | |
| (i) Parent rock | ... | 44.1 | .... | 16.9 | 7.5 | 1.8 | 1.2 | 14.5 | 7.7 | 4.8 | .... | 1.4 | 4.42 | 0.07 | 9.42 | 2.1 |
| (ii) Laterite | 2.8 | 15.5 | 24.9 | 11.3 | 13.0 | 10.4 | 2.5 | (1.4) | | ... | 13.2 | 4.8‡ | 2.32 | 0.59 | 0.97 | 0.42 |
| (iii) Pink earth | 1.1 | 33.5 | 3.0 | 29.3 | 9.3 | 8.6 | 1.1 | (0.7) | | ... | 1.6 | 11.4‡ | 1.94 | 0.19 | 0.88 | 0.45 |
| (iv) Red earth | 2.8 | 30.0 | 3.3 | 27.1 | 8.7 | 10.6 | 1.3 | 0.3 | 2.2 | ... | 1.7 | 11.0‡ | 1.89 | 0.25 | 0.82 | 0.43 |
| (v) Surface soil | 3.3 | 29.0 | 2.0 | 22.3 | 6.9 | 15.4 | (1.9) | 0.4 | 2.5 | ... | 1.1 | 11.1‡ | 2.20 | 0.44 | 0.78 | 0.36 |
| *Colloidal clay* | | | | | | | | | | | | | | | | |
| (ii) Laterite | 0.3 | 25.4 | 26.5 | 7.1 | .... | 22.3 | 1.0 | .... | ... | ... | 14.0 | 3.1 | (6.04) | 2.00 | 2.46 | 0.41 |
| (iii) Pink earth | 0.1 | 38.3 | 9.5 | 19.1 | .... | 16.6 | 0.8 | .... | ... | ... | 5.0 | 9.4 | (3.41) | 0.55 | 1.22 | 0.36 |
| (iv) Red earth | 3.5 | 34.1 | 6.1 | 28.0 | .... | 10.5 | 0.8 | .... | ... | ... | 3.2 | 12.4 | 2.07 | 0.24 | 0.82 | 0.40 |
| (v) Surface soil | 1.2 | 35.8 | 3.7 | 29.9 | .... | 12.5 | 1.1 | .... | ... | ... | 2.0 | 13.6 | 2.04 | 0.27 | 0.79 | 0.39 |
| *"Ideal" clay-minerals* | | | | | | | | | | | | | | | | |
| Gibbsite ($Al_2O_3 \cdot 3H_2O$) | | | | | | | | | | | | | .... | .... | .... | 0.33 |
| Kaolinite ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$) | | | | | | | | | | | | | 2.00 | .... | 1.00 | 0.50 |
| Halloysite ($Al_2O_3 \cdot 2SiO_2 \cdot 4H_2O$) | | | | | | | | | | | | | 2.00 | .... | 0.50 | 0.25 |
| Beidellite ($Al_2O_3 \cdot 3SiO_2 \cdot 3H_2O$) | | | | | | | | | | | | | 3.00 | .... | 1.00 | 0.33 |
| Nontronite ($Fe_2O_3 \cdot 4SiO_2 \cdot 2H_2O$) | | | | | | | | | | | | | .... | .... | 0.50 | .... |

* Combined components.

† Expressed as $Fe_2O_3$ but containing 6.6 per cent $TiO_2$.

‡ Corrected for organic matter.

a powerful electromagnet, and weighing. In the three sand grades, the remainder was then settled in a solution of potassium mercuric iodide ($KHgI_3$; sp.gr. 2.94) and the proportion of heavy residue estimated by weighing. The identity of the minerals comprising both light and heavy fractions was deter-

TABLE 2

*Transformations between parent rock and earthy products*

Compositions recalculated to constant total alumina

| | PERCENTAGE COMPOSITION | | | | | LOSSES OR GAINS | | | |
|---|---|---|---|---|---|---|---|---|---|
| | (i) Rock | (ii) Laterite | (iii) Pink earth | (iv) Red earth | (v) Soil | (ii) Laterite | (iii) Pink earth | (iv) Red earth | (v) Soil |
| | | *Whole material* | | | | | | | |
| Quartz | ..... | 1.3 | 0.6 | 1.6 | 2.3 | +1.3 | −0.7 | +1.0 | +0.7 |
| Combined $SiO_2$ | 44.1 | 7.2 | 17.5 | 16.7 | 20.2 | −36.9 | +10.3 | −0.8 | +3.5 |
| Gibbsite | ..... | 11.6 | 1.6 | 1.8 | 1.4 | +11.6 | −10.0 | +0.2 | −0.4 |
| Combined $Al_2O_3$ | 16.9 | 5.3 | 15.3 | 15.1 | 15.5 | −11.6 | +10.0 | −0.2 | +0.4 |
| Magnetite | 7.5 | 6.1 | 4.9 | 4.8 | 4.8 | −1.4 | −1.2 | −0.1 | 0.0 |
| Amorphous $Fe_2O_3$ | 1.8 | 4.9 | 4.5 | 5.9 | 10.7 | +3.1 | −0.4 | +1.4 | +4.8 |
| $TiO_2$ | 1.2 | 1.2 | 0.6 | 0.7 | 1.3 | 0.0 | −0.6 | +0.1 | +0.7 |
| CaO | 14.5 | 0.6 | 0.4 | 0.2 | 0.3 | −21.6 | −0.2 | +1.0 | +0.1 |
| MgO | 7.7 | | | 1.2 | 1.7 | | | | +0.5 |
| $K_2O + Na_2O$ | 4.8 | ..... | ..... | ..... | ..... | ..... | ..... | ..... | ..... |
| $H_2O$ | | | | | | | | | |
| Gibbsitic | ..... | 6.2 | 0.8 | 0.9 | 0.8 | +6.2 | −5.4 | +0.1 | −0.1 |
| Other | 1.4 | 2.2 | 6.0 | 6.1 | 7.7 | +0.8 | +3.8 | +0.1 | +1.6 |
| Totals | 99.9 | 46.6 | 52.2 | 55.0 | 66.7 | −53.3 | +5.6 | +2.8 | +11.7 |
| | | *Colloidal clay* | | | | | | | |
| Quartz | ..... | 0.1 | 0.1 | 1.7 | 0.6 | +0.1 | 0.0 | +1.6 | −1.1 |
| Combined $SiO_2$ | 44.1 | 12.8 | 22.6 | 16.9 | 18.2 | −31.3 | +9.8 | −5.7 | +1.3 |
| Gibbsite | ..... | 13.3 | 5.6 | 3.0 | 4.3 | +13.3 | −7.7 | −2.6 | +1.3 |
| Combined $Al_2O_3$ | 16.9 | 3.6 | 11.3 | 13.9 | 12.6 | −13.3 | +7.7 | +2.6 | −1.3 |
| $Fe_2O_3$ | 9.3 | 11.2 | 9.8 | 5.2 | 6.4 | +1.9 | −1.4 | −4.6 | +1.4 |
| $TiO_2$ | 1.2 | 0.5 | 0.5 | 0.4 | 0.6 | −0.7 | 0.0 | −0.1 | +0.2 |
| $H_2O$ | | | | | | | | | |
| Gibbsitic | ..... | 7.0 | 3.0 | 1.6 | 2.2 | +7.0 | −4.0 | −1.4 | +0.6 |
| Other | 1.4 | 1.6 | 5.6 | 6.1 | 5.7 | +0.2 | +4.0 | +0.5 | −0.4 |
| Totals | 72.9 | 50.1 | 58.5 | 48.8 | 50.6 | −22.8 | +8.4 | −9.7 | +1.8 |

mined under the microscope by simple optical tests and, in some cases, by dye-stuff tests. The results are presented in table 4. Although the materials show considerable variation in texture (the content of the finer fractions increasing from the laterite to the red earth), the mineralogical composition is remarkably uniform, not only in the different grades, but also in the three

TABLE 3

*Transformations between parent rock and earthy products*

Compositions recalculated to kaolinite

| | PERCENTAGE COMPOSITION | | | | | LOSSES OR GAINS | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Kaolinite (ideal formula) | (ii) Laterite | (iii) Pink earth | (iv) Red earth | (v) Soil | (ii) Laterite | (iii) Pink earth | (iv) Red earth | (v) Soil |
| | | *Whole material* | | | | | | | |
| Nonquartz $SiO_2$ | 46.6 | 54.0 | 45.0 | 43.7 | 51.2 | +7.4 | −1.6 | −2.9 | +4.6 |
| Combined $Al_2O_3$ | 39.4 | 39.4 | 39.4 | 39.4 | 39.4 | ..... | ..... | ..... | ..... |
| $H_2O$ | 14.0 | 16.7 | 15.3 | 16.0* | 19.6* | +2.7 | +1.3 | +2.0 | +5.6 |
| | | *Colloidal clay* | | | | | | | |
| (Clay ..... *per cent* | ..... | 10.1 | 20.1 | 47.5 | 50.0) | | | | |
| Nonquartz $SiO_2$ | 46.6 | *141.0* | *79.0* | 48.0 | 47.2 | *+94.4* | +32.4 | +1.4 | +10.3 |
| Combined $Al_2O_3$ | 39.4 | 39.4 | 39.4 | 39.4 | 39.4 | ..... | ..... | ..... | ..... |
| $H_2O$ | 14.0 | 17.2 | 19.4 | 16.7* | 17.8* | +3.2 | +5.4 | +2.7 | +3.8 |
| | | *Sand + silt* | | | | | | | |
| Nonquartz $SiO_2$ | 46.6 | 48.3 | 39.9 | 39.4 | 59.5 | +1.7 | −6.7 | −7.2 | −2.4 |
| Combined $Al_2O_3$ | 39.4 | 39.4 | 39.4 | 39.4 | 39.4 | ..... | ..... | ..... | ..... |
| $H_2O$ | 14.0 | 16.3 | 14.7 | 15.1* | 23.2* | +2.3 | +0.7 | +1.1 | +7.9 |
| *Computation as minerals* (assuming kaolinite formation) | | *Whole material* | | | | *Colloidal clay* | | | |
| *Crystalline components* | | | | | | | | | |
| Gibbsite | | 38.1 | 4.6 | 5.0 | 3.1 | 40.5 | 14.5 | 9.3 | 5.7 |
| Kaolinite | | 28.6 | 74.3 | 68.5 | 56.4 | 18.0 | 48.3 | 70.8 | 75.6 |
| Quartz | | 2.8 | 1.1 | 2.8 | 3.3 | 0.3 | 0.1 | 3.5 | 1.2 |
| Magnetite | | 12.6 | 9.0 | 8.4 | 6.7 | ..... | ..... | ..... | ..... |
| *Amorphous components* | | | | | | | | | |
| Free iron oxides | | 10.7 | 8.9 | 10.9 | 15.6 | 22.3 | 16.6 | 10.5 | 12.5 |
| Free silica | | 2.2 | 0 | 0 | 2.7 | *17.0* | *15.8* | 1.2 | 0.6 |
| Titania | | 2.5 | 1.1 | 1.3 | 1.9 | 1.0 | 0.8 | 0.8 | 1.1 |
| Bases | | 1.4 | 0.7 | 2.5 | 2.9 | ..... | ..... | ..... | ..... |
| Water (excess) | | 0.8 | 1.1 | 1.5 | 3.3 | 0.6 | 2.7 | 2.5 | 3.1 |

*Corrected for organic matter contents.

different profile materials. The bulk of the mineral matter resembles kaolinite in its optical properties.

### *Dehydration curves*

Samples of the fine silt and clay fractions of the laterite, pink earth, and red earth and of gibbsite were exposed to a moist atmosphere in a humidifier con-

taining 50 per cent sulfuric acid in order to bring their initial moisture contents into comparable states. Weighed amounts were then heated in stages to 260°C. in silica weighing bottles in a thermostatically regulated electric oven, and afterward to 800°C. in an electric muffle furnace. The losses in weight for different stages in the heating were determined, and graphs were constructed from the results (fig. 1). Dehydration data for kaolinite, halloysite, and

TABLE 4

*Mineralogical composition of red earth profile*

Materials washed with boiling acid before mineralogical examination*

| | | (i) PARENT ROCK | (ii) LATERITE CRUST | (iii) PINK EARTH | (iv) RED EARTH |
|---|---|---|---|---|---|
| | | *per cent* | *per cent* | *per cent* | *per cent* |
| *Texture analysis* | | | | | |
| Coarse sand.....(>200 μ) | | .... | 22.3 | 31.4 | 18.4 |
| Medium sand.....(200–50 μ) | | 67.6 | 28.3 | 20.5 | 19.7 |
| Fine sand.....(50–10 μ) | | 19.8 | 0.0 | 17.2 | 12.3 |
| Coarse silt.....(10–5 μ) | | 9.7 | 34.9 | 0.0 | 9.0 |
| Fine silt + clay.....(<5 μ) | | 2.9 | 14.5 | 30.9 | 40.6 |
| *Mineral separation* (Heavy liquid: sp. gr. 2.94) | | | | | |
| Coarse sand: | Magnetite..... | .... | 25.3 | 13.4 | 24.4 |
| | Heavy residue... | .... | 0.7 | 0.0 | 12.4 |
| | Light fraction... | .... | 74.0 | 86.6 | 63.2 |
| Medium sand: | Magnetite..... | 7.6 | 23.2 | 22.1 | 23.6 |
| | Heavy residue... | 18.2 | 1.1 | 6.8 | 12.4 |
| | Light fraction... | 74.2 | 75.7 | 71.1 | 64.0 |
| Fine sand: | Magnetite..... | 8.3 | .... | 1.6 | 0.0 |
| | Heavy residue... | 18.0 | .... | 0.0 | 0.8 |
| | Light fraction... | 73.7 | .... | 98.4 | 99.2 |
| Notes Heavy residue..... | | Augite mainly | Trace of augite only | Iron oxide concretions mainly | Iron oxide concretions |
| Light fraction..... | | Plagioclase felspar mainly | Much kaolinitic mineral | Kaolinitic mineral mainly | Kaolinitic mineral mainly |

* Parent rock ground, other profile parts crushed.

beidellite, published by Kelley, Jenny, and Brown (12), were also plotted for comparison on the same diagram.

The curves for the three different materials of the red earth profile show remarkable similarity to the curve for halloysite, although they also show some resemblance to that for kaolinite. The laterite curve is obviously modified by its content of gibbsite.

*Physical and chemical soil analysis*

Determinations of some constants were made by standard routine methods. The results are set out in table 5. The main features of the profile materials

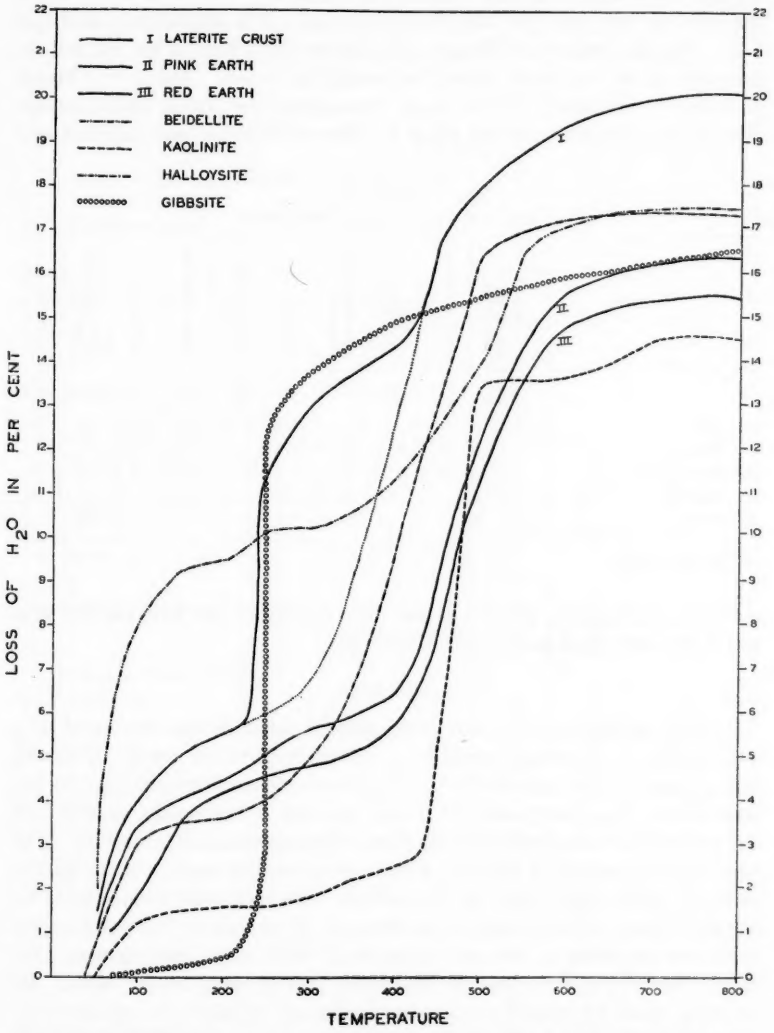


FIG. 1. DEHYDRATION CURVES FOR FINE SILT AND CLAY FRACTIONS OF LATERITE, PINK EARTH, AND RED EARTH PARTS OF PROFILE AND FOR SEVERAL MINERALS

In plotting the gibbsite curve the actual figures obtained for percentage water loss have been halved

are the fair constancy but appreciable magnitude of the values for base-exchange capacity (average, 22.6 m.e.), for sticky point moisture (41.3 per cent), and for residual shrinkage (4.4 per cent), denoting the presence of a mineral resembling halloysite rather than kaolinite. The three layers above the rock are very acid (pH 4.5), but the surface soil is only slightly acid (pH 6.2). Organic matter and nitrogen contents are fairly high in the soil and appreciable in the red earth layer, but negligible below. Available (Truog) phosphate is uniformly low (18 p.p.m.) throughout the profile, and exchangeable potash is medium-low (94 p.p.m.). The soil is not so well supplied with

TABLE 5

*Some soil constants of red earth profile*

| MATERIAL | STICKY POINT MOISTURE | TOTAL SHRINKAGE | RESIDUAL SHRINKAGE | BASE EXCHANGE CAPACITY | REACTION | TOTAL ORGANIC MATTER | TOTAL NITROGEN | C/N RATIO | AVAILABLE $P_2O_5$ (TRUOG) | AVAILABLE (EXCH.) $K_2O$ |
|---|---|---|---|---|---|---|---|---|---|---|
| | *per cent* | *per cent** | *per cent** | *m.e.* | *pH* | *per cent* | *per cent* | | *p.p.m.* | *p.p.m.* |
| (v) Soil | 41.3 | .... | ... | .... | 6.2 | 4.0 | 0.22 | 10.8 | 22 | 92 |
| (iv) Red earth | 42.0 | 46.1 | 4.3 | 24.4 | 4.4 | 1.1 | 0.06 | 10.8 | 13 | .. |
| (iii) Pink earth | 41.6 | 24.8 | 4.5 | 21.3 | 4.3 | 0.3 | 0.02 | 9.3 | 16 | 96 |
| (ii) Laterite | 40.4 | .... | ... | 22.2 | 4.7 | 0.1 | 0.01 | 7.3 | 21 | 60 |
| (i) Rock | .... | .... | ... | .... | 7.9 | ... | .... | .... | 66 | .. |

* Of dry volume.

nutrients, particularly potash, as are the majority of the Grenada red soils which regularly yield good crops of cacao (8).

### *Dye tests*

*Alizarin* adsorption for determining gibbsitic alumina was measured by a modification of the earlier method (5), which involves the use of an electric furnace rather than uncontrolled heating in crucibles over open burners (*see* appendix). The alizarin-stained *ignited* material is thoroughly washed with hot water rather than boric alcohol before stripping with oxalate solution. The alizarin dye-standard is made up with a blank oxalate extract of the ignited material, rather than pure oxalate solution; this accurately compensates for ferrioxalate ion, which imparts an undesirable green color, so that no blue dye need now be added to the color standard. With these improvements, the alizarin method is considered to be reliable and quantitatively precise. On the other hand, its value for estimating hydrous iron oxides in *unignited* fresh materials still remains doubtful, since certain clay minerals (for example, halloysite and beidellite) have been found capable also of adsorbing alizarin before ignition (*see* table 6).

The adsorption of dyes other than alizarin was examined as a possible means

TABLE 6

*Comparative dye tests on standard materials and red earth profile samples*

Values in dye units absorbed per gram of air-dry material

| MATERIALS* | LOSS ON IGNITION | ALIZARIN UPTAKE | DIAMINE SKY BLUE UPTAKE | | | JANUS RED UPTAKE |
|---|---|---|---|---|---|---|
| | | | Boric | Phosphatic | Difference | |
| | *per cent* | | | | | |
| *Authentic minerals* | | | | | | |
| 1. "New" aluminum hydroxide... | 44.9 | 1250 (2273)† | 1112 (2018) | 12 (22) | 1100 (1996) | 10 |
| 2. "Aged" aluminum hydroxide... | 55.2 | 665 (1486) | 76 (170) | 82 (183) | 0 (0) | 18 |
| 3. Gibbsitic bauxite..... | 33.1 | 4 (6) | 93 (150) | ..... | ..... | 6 |
| 4. Ferric hydroxide..... | 44.8 | 1375 (2490) | 2544 (4610) | 0 (0) | 2544 (4610) | 6 |
| 5. Turgite..... | 17.0 | 168 (213) | 328 (416) | 0 (0) | 328 (416) | 20 |
| 6. Limonite..... | 10.8 | 38 (46) | 46 (56) | ..... | ..... | 47 |
| 7. Kaolinite..... | 12.8 | 15 | 52 (60) | 50 (57) | 2 (3) | 340 |
| 8. Halloysite..... | 17.2 | 148 | 667 (805) | 517 (624) | 150 (181) | 1240 |
| 9. Beidellite..... | 20.6 | 93 | 80 (101) | ..... | ..... | 982 |
| 10. Montmorillonite..... | 19.2 | 11 | 31 (38) | 30 (37) | 1 (1) | 2000 |
| *Profile samples—whole material* | | | | | | |
| (i) Parent rock..... | 1.4 | 21 | 30 (31) | 25 (25) | 0‡ (0) | 83 |
| (ii) Laterite..... | 18.0 | 99 | 390 (473) | 83 (100) | 282‡ (344) | 704 |
| (iii) Pink earth..... | 13.0 | 147 | 574 (644) | 246 (286) | 254‡ (292) | 1298 |
| (iv) Red earth..... | 12.7 | 143 | 482 (552) | 85 (97) | 371‡ (425) | 1096 |
| (v) Soil..... | 12.2 | 107 | 60 (69) | 40 (45) | 8‡ (9) | 1056 |
| *Profile samples—colloidal clay* | | | | | | |
| (ii) Laterite clay..... | 17.1 | ..... | 1040 (1250) | 310 (370) | 668‡ (806) | 790 |
| (iii) Pink earth clay..... | 14.4 | ..... | 815 (950) | 465 (545) | 257‡ (300) | 2365 |

* All materials previously air-dried, but *not* ignited, and ground to pass 100-mesh sieve.

† Figures in parentheses are dye values recalculated to ignited basis.

‡ Dye values corrected for dye uptake by phosphated halloysite.

TABLE 6—*Concluded*

*Sources of minerals:*

1, 2, 4. *Laboratory preparations;* precipitation from solutions of metal-chloride by ammonium hydroxide at 0°C. "Aged" alumina, stored for 8 years.

3. *Gibbsitic bauxite;* Demerara Bauxite Co., high-grade ore. Contains 62 per cent $Al_2O_3$ and 33 per cent $H_2O$.

5. *Turgite;* Gregory, London. Contains 79 per cent $Fe_2O_3$ and 17 per cent $H_2O$.

6. *Limonite;* Eimer and Amend, New York. Contains 82 per cent $Fe_2O_3$ and 11 per cent water.

7. *Kaolinite;* Baird and Tatlock, London. Commercial sample.

8. *Halloysite;* Lawrence, Missouri. Presented by Dr. C. E. Marshall.

9. *Beidellite;* Clay fraction of Putnam clay. Presented by Dr. C. E. Marshall.

10. *Montmorillonite;* Clay fraction of Volclay. Presented by American Colloid Co. by request of Dr. C. E. Marshall.

of identifying and approximately estimating specific clay minerals as well as hydrous iron oxides. Some 30 different dyes were tried, including examples of basic and acidic classes, but only two—diamine sky blue and janus red—have so far proved useful. The methods of applying them are given in the appendix. The results obtained by dye tests, both for standard minerals and for red earth profile samples, are set out in table 6.

## RESULTS

### *Laterite formation*

The analytical data in tables 1 and 2 clearly demonstrate the nature of the profound alterations that affect the parent rock, changing it into primary laterite. Assuming no loss in total alumina, the chief results are (*a*) the liberation and removal of nearly all the bases (potash, soda, lime, magnesia), (*b*) the liberation of combined silica, much of which is evidently transported upward into the layer above, though a little crystallizes as secondary quartz, most of the remainder being lost, (*c*) the liberation and crystallization as gibbsite of a large part of the combined alumina, (*d*) the change of some of the magnetite into hydrous iron oxide, (*e*) the formation of hydrous iron oxide by the hydrolysis of the ferromagnesian minerals, (*f*) the absorption of a considerable quantity of water, part of which is fixed in new crystalline minerals, and part in colloidal hydrous oxides. These transformations are very similar to those described by Harrison (9) and by Hardy and Follett-Smith (7) as affecting basic igneous rocks in British Guiana.

The total loss of oxide components (mainly silica and bases) by the decomposing parent rock in the Grenada profile amounts to over 60 per cent, and the first product (primary laterite) is distinguished by the occurrence of the unique secondary mineral, gibbsite ($Al_2O_3 \cdot 3H_2O$), comprising nearly 70 per cent of its total alumina content.

The remainder of the alumina, assuming no removal, has presumably combined with part of the liberated silica to form some new aluminosiliceous mineral, the identity of which needs to be determined now. The evidence sup-

plied by chemical analysis alone is not sufficient for complete identification, but information provided by mineralogical examination, by physical constants, and particularly by the striking dehydration curves, proves beyond doubt that it is a mineral of the kaolin class, resembling halloysite in most of its properties, although its content of constitutional water falls short of the requirement of the formula, $Al_2O_3 \cdot 2SiO_2 \cdot 4H_2O$, rather suggesting *metahalloysite*, ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$) (14). Assuming the formula for metahalloysite, the computations show that the primary laterite contains about 38 per cent gibbsite, 29 per cent metahalloysite, 13 per cent magnetite, and 3 per cent secondary quartz, a total of 83 per cent crystalline components. The residue is apparently made up of 2½ per cent titania, 1 per cent bases, 1 per cent combined water, 10½ per cent free iron oxide, and 2 per cent silica. The occurrence in the laterite of unaccounted and presumably free silica other than quartz is significant, and the analytical data prove that it is confined almost entirely to the colloidal clay fraction (table 3). This fraction, comprising 10 per cent of the whole material, apparently contains no less than 17 per cent unaccounted silica not quartz, if metahalloysite is assumed to be the only aluminosiliceous mineral present. As there is no evidence that this surplus silica occurs in any other combination (such as anauxite, allophane, pyrophyllite, or nontronite), it may be regarded as amorphous or colloidal silica that has been liberated but not removed. It may comprise the chief cementing agent that confers brittleness on the coherent laterite crusts.

The hydrous iron oxide residues, other than those derived from magnetite, have remained as pseudomorphs of the augite phenocrysts[2] and have not migrated perceptibly into the groundmass of the laterite.

### *Pink earth formation*

The layer above the laterite crusts covering decomposing basic igneous rock, and presumably derived from it, is remarkable for the fact that its content of gibbsite is very much less and its content of combined alumina is correspondingly greater than that of the laterite.[3]

[2] Analyses of porphyritic augite crystals isolated from the Grenada augite-andesite recorded by Harrison (10) show: $SiO_2$, 46.8; $Al_2O_3$, 8.8; $Fe_2O_3$, 10.5; CaO, 20.9; MgO, 13.3 per cent (total 100.3). Hence the partition of the oxide components in the augite contained in the parent rock (15.5 per cent) should be, $SiO_2$, 7.3; $Al_2O_3$, 1.4; $Fe_2O_3$, 1.7; CaO, 3.2; MgO, 2.1 per cent, leaving $SiO_2$, 36.8; $Al_2O_3$, 15.5; $Fe_2O_3$, 0.1; CaO, 11.3; MgO, 5.6 per cent to be shared between plagioclase felspars and olivine. As the small residue (0.1) of $Fe_2O_3$ implies that the olivine is a magnesic and not a ferric type (i.e. forsterite), most of the iron oxide residues resulting from the weathering of this rock are derived from its augite component, thus explaining the relatively pale color of the groundmass of the laterite crust and of the pink earth.

[3] Harrison (9) explains this striking feature by supposing that *resilication* of gibbsite proceeds in the upper part of the laterite layer as a result of the upward movement of groundwater carrying colloidal silica or alkali silicates in solution. He further supposes that this rising of ground water occurs by capillarity during dry seasons at sites where the drainage is impeded. Harrison assumes that, under conditions of free drainage, no resilication proceeds, but instead, the primary laterite accumulates in increasing thicknesses.

The product of gibbsite silication is believed to be some *kaolin* mineral, and the analytical data, augmented by other evidence presented in the tables and the dehydration graphs, support this conclusion in the case of the Grenada profile and suggest further that it is metahalloysite, as in the laterite. An appreciable residue (16 per cent) of uncombined silica remains in the clay fraction of the pink earth (table 3), as in the laterite, although it is absent from the overlying red earth.

The pink earth contains 74 per cent metahalloysite, 5 per cent gibbsite, 9 per cent magnetite, and 1 per cent quartz, a total of 89 per cent crystalline components. The amorphous residue consists mainly of 9 per cent free iron oxide (presumably hydrated by the 1 per cent unaccounted combined water), which is very unevenly distributed within the gray-white kaolinitic clay and still exhibits marked segregation as augite pseudomorphs. The pink earth evidently comprises an *active zone of secondary changes*, and represents a transition between gibbsitic laterite and red earth proper.

### *Red earth formation*

The red earth layer is a homogeneous mass apparently consisting chiefly of metahalloysite (68½ per cent), stained uniformly red by hydrous ferric oxide (10½ per cent), together with 5 per cent gibbsite, 9 per cent magnetite, and 3 per cent secondary quartz. The composition of its colloidal clay component, comprising about 50 per cent of the whole, is almost identical with that of the rest of the material, a noteworthy feature of the red earth. Furthermore, the clay contains no excess of free colloidal silica, the resilication of gibbsite evidently having proceeded as far as possible at the base of the layer. The red earth proper may thus be regarded as a *zone of completed secondary changes*.

As organic matter occurs in appreciable quantity (1.1 per cent; *see* table 5) in the red earth layer, it evidently comprises part of the solum. Plant roots occur throughout its depth, some even penetrating the pink earth below and reaching the laterite-encrusted boulders where these are not too deep-seated. The mineralization of the organic matter may account for the somewhat greater content of bases in the red earth as compared with the underlying pink earth.

### *Surface soil formation*

The uppermost layer of the Grenada profile differs only slightly in appearance from the layer below, which it closely resembles in chemical and mineralogical composition. Its crystalline components consist of 56 per cent metahalloysite, 3 per cent gibbsite, 3 per cent quartz, and 7 per cent magnetite; its amorphous part, amounting to 30 per cent of the whole, mainly comprises hydrous iron oxide (15½ per cent). It contains more total bases and constitutional water than does the red earth layer, and it shows a relatively higher content of titania, considered to be a characteristic feature of the A horizon of lateritic types of soil as contrasted with podzolic types (11). The

clay fraction of the soil includes a higher proportion of kaolinitic mineral than the sand and silt, but less free quartz.

These results do not demonstrate the effects of profound leaching (tropical podzolization), such as prevails in British Guiana where red earths usually exhibit bleached sandy surface layers, consisting almost entirely of white residual quartz. Nowhere in Grenada is such highly leached degraded red earth to be found, the soils instead appear to be somewhat immature, and in certain cases, the composition of their surface layers has apparently been modified by a covering of geologically recent volcanic ash derived within the island itself or from some neighboring island.

## DISCUSSION

### *Primary laterization*

Primary laterization appears to be a fundamental process, apparently dependent on the conditions of weathering[4] as well as on the chemical or mineralogical composition of the parent rock.[5] Abundant evidence may be adduced in support of the view that acidic igneous rocks, rich in 6-silica feldspars, microcline, and quartz, when hydrolyzed, liberate such large quantities of reactive silica (colloidal silica or silicate ion), that alumina molecules immediately on liberation, are silicated into a secondary kaolinitic mineral or even sericite mica, free alumina seldom appearing among the end products. On the other hand, basic igneous rocks, containing 2-silica feldspars (lime feldspars) and little or no quartz, liberate much lesser amounts of reactive silica in proportion to alumina, and consequently, in accordance with the laws of mass action, much lesser amounts of kaolinitic products appear, the excess of free alumina crystallizing out as gibbsite. Presumably the formation and accumulation of gibbsite would be enhanced by free drainage, because this favors the rapid removal of silica in solution. Harrison repeatedly stresses the importance of free drainage in primary laterization, and it is generally agreed that kaolinization proceeds best under poor drainage conditions.

### *Silication of gibbsite*

The resilication theory, as postulated by Harrison (9), would probably not be accepted by physical chemists, although the essential facts now appear to be demonstrable. The conditions favoring the process have not been specifically investigated, but merely conjectured. Resilication necessitates, first, the provision of an abundant and continuous supply of silica or silicate ion in solution and, secondly, its transportation to and precipitation within the mass of gibbsite-containing laterite. The coincidence of these two circumstances would appear to be rare, since the greatest amount of silica is liberated through mineral hydrolysis during the wet season when the rainfall is continuous and heavy and when water is con-

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[4] For example, Mattson (16) regards laterization as essentially a mineral hydrolysis proceeding under alkaline conditions.

[5] De Sigmond (19, p. 29) refers to his researches on artificial zeolites, by which he demonstrated that "in an alkaline medium, $Al_2O_3$ is usually precipitated together with a part of the silicic acid and with some of the bases, while the rest of the silicic acid and bases remains in solution and may be leached out. This occurs during the weathering of natural silicates in pure water. In an acid medium, different conditions prevail." He also (19, p. 23) cites Harrassowitz in support of the view that, "whereas in orthoclase, kaolin may really be formed, the products resulting from calcium and sodium felspars are allophanoids" (that is, mutual solutions of silica, alumina, and water, easily dissolved by hydrochloric acid, and colloidal in form).

stantly moving *downward*, whereas the process of resilication presumably necessitates, according to Harrison, *upward* movement of ground water. Harrison assumes that this occurs as a result of capillary action during the dry season. Actually, at this period, there may be an insufficiency of colloidal silica to effect resilication, because most of the silica-bearing water may already have drained away.

It is possible, however, to explain gibbsite silication in another way, namely, by taking into account the effects of *dilution* of the concentrated solution of colloidal silica by *descending* water that has penetrated the soil as rain.[6] This water has already traversed a highly acidic layer of red earth before intermingling with the solution of silica; its reaction, therefore, is probably acidic. Furthermore, mere dilution has been shown by Buehrer and Williams (2) greatly to reduce the alkalinity of mixtures of finely divided feldspars and pure water, although no explanation is offered of the phenomenon. Thus, in the case of albite, a decrease in pH value from 9.5 to 6.2 was observed for a change in feldspar-water ratio of 1:2 to 1:100.

The new theory may be stated as follows: We may first assume that the laterite layer is impregnated from below with a concentrated alkaline solution of colloidal silica throughout the wet season, and that excess of solution is continually draining away over the decomposing rock surface. We may further assume that the level of saturation falls rapidly in the intervals between rainy spells, and during these spells the upper part of the solution is subjected to dilution by descending acidic water. Under these circumstances, silica will be precipitated around the gibbsitic nuclei occurring in the upper part of the laterite layer. The marked change in reaction conditions in this part of the profile will favor the mutual adsorption of alumina and silica, for, according to the isoelectric theory as postulated by Mattson (16), the alumina particles will acquire electropositive charges when the reaction becomes more acidic than the isoelectric point of alumina (stated variously to range from pH 6.5 to 8.1), whereas the silica particles will be electronegatively charged, having an isoelectric point very low on the pH scale. By slow degrees, the coprecipitate will become stabilized through irreversible dehydration, favored especially by the onset of the dry season when the water table sinks to the level of the parent rock. It will slowly "age," passing through stages such as allophane, until it eventually crystallizes as a kaolinitic mineral, probably halloysite.

It seems likely, therefore, that both primary laterization and resilication occur across narrow boundary zones, and that reaction conditions, as well as degree of wetness and solute concentration, may fluctuate constantly between wide limits, setting up reversible gradients alternately favoring the one process and then the other.

The mechanism of laterization and gibbsite silication may be complicated in some instances by changes in ground-water conditions consequent on a lowering of base levels of erosion by tectonic uplift, which may result in a rejuvenation of the drainage.[7] Such changes might favor laterization rather than kaolinization; consequently, a zone of primary laterization displayed at the present time in a red earth profile may merely represent a comparatively recent development. A historic sequence of topographical events similar to this actually appears to have affected the hinterland of British Guiana during late Pleistocene times, and evidence is afforded by the occurrence of high-level limestones that Grenada also was elevated above the sea during the same geological period. On the other hand, red earth derived from basalt in South Carolina (Davidson clay loam)[8] has been proved by the writers to exhibit very similar features to those of the Grenada soil, clearly showing a transition between primary gibbsitic laterite and kaolinic red earth. Whether the incidence of successive phases

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[6] The writers are indebted to Irvin C. Brown, of the U. S. Bureau of Soils, for this suggestion, communicated by G. Milne.

[7] G. Milne, private communication.

[8] The writers are indebted to J. F. Lutz, of the North Carolina State College of Agriculture, for providing profile samples of the Davidson red earth, including laterite crusts on basalt boulders.

of a changing set of geological conditions must be invoked to explain the development of red earth in the southeastern United States, as well as in the other two areas, could only be decided by further intensive investigations.

*Redistribution of iron oxide in the pink earth layer*

The most striking difference between the red earth and the pink earth is its uniform bright coloration, as contrasted with the orange-mottled, fine red-speckled, and patched bluish-gray appearance of the pink earth. This cannot be accounted for by a difference in gross composition; it is apparently solely due to a more uniform distribution of the iron oxide component in the red earth. Possibly iron occurs in the pink earth partly in the ferrous state, generally believed to be more mobile than ferric iron. The constant wetness of the material would be conducive to the maintenance of temporary reducing conditions, under which some of the iron compounds would exhibit bluish colorations. During upward diffusion into a drier zone, rapid oxidation of iron and the partial dehydration and agglomeration of its hydrous oxide products might produce a stable red mineral resembling goethite ($Fe_2O_3 \cdot H_2O$) or a mixture of goethite and hematite ($Fe_2O_3$). Whether the diffusion or migration of the hydrous ferric oxides is aided by colloidal silica (the occurrence of which in the pink earth is indicated by the chemical analyses) in accordance with the theory of Riefenberg (17), or by some other agency such as humus, can only be conjectured as possibilities. Whereas the pink earth may be regarded as a zone of intermittent saturation within which active secondary changes proceed, the red earth layer may be considered to be a zone of non-saturation where these changes are completed and where equalization, fixation, and thorough oxidation of the iron oxide component are effected. The red earth is the end product of this sequence of mineralogical transformations; to the pedologist it would therefore comprise the parent material from which a fertile soil might be produced by the cooperation of biotic factors.

*Identity and properties of the iron oxide component*

The most characteristic and interesting component of the red earth profile is hydrous iron oxide, the properties of which probably affect greatly the agricultural behavior of the soil when cropped, as well as its response to manuring. Its main feature appears to be a high specific adsorptive capacity for acidic radicals or colloidal anions, as revealed, for example, by its pronounced uptake of acidic dyes such as diamine sky blue (table 6). For purposes of comparison, dye values for various specimens of alumina, iron oxide, and clay minerals were determined. The results show that the more highly hydrated iron oxides and fresh aluminum hydroxide strongly adsorb alizarin and diamine sky blue from their solutions in boric alcohol, though these dyes are also appreciably adsorbed by halloysite, only slightly by beidellite, and scarcely at all by gibbsite, kaolinite, and montmorillonite. In the presence of phosphate, however, hydrous iron oxides and aluminum hydroxide no longer adsorb diamine sky blue (a blocking effect by phosphate), but halloysite and beidellite still adsorb it, showing a diminution of only 30 per cent in dye value. On the other hand, hydrous iron oxides and aluminum hydroxide do not appreciably adsorb janus red (a basic dye), nor does gibbsite, but still the clay minerals adsorb it very strongly, kaolinite least, and montmorillonite most. Unfortunately, the magnitudes of the dye values cannot be regarded as quantitatively significant, because they depend on several uncontrolled variable factors, notably particle size, degree of hydration, and state of agglomeration. Partly for this reason, no attempt will be made to interpret the results for the profile layers except in a general way.

From the diamine sky blue data in table 6, it will be noted that the material of each of the layers except the surface soil apparently contains considerable amounts of free iron oxide. The average dye value, per gram $Fe_2O_3$, is 3,570 units, which further suggests that this component is an extremely active adsorbent, resembling the artificial preparation of ferric hydroxide (4,610 units per gram $Fe_2O_3$) rather than either of the specific minerals, turgite and

limonite. This finding agrees with the results of the previous investigation of iron oxide minerals by the original alizarin method (7).

The iron oxide component of the surface soil, although greater in quantity than that of the layers below, is evidently very much less reactive, its dye value, per gram $Fe_2O_3$, being only 60 units. This may imply a much lower degree of hydration (insofar as hydration may partly decide the absorbing capacity of hydrous iron oxides for acidic dyes), indicating the pronounced surface drying to which the soil is subjected during dry seasons.

From the results of the janus red tests, it will be seen that the material of each layer contains large amounts of a reactive aluminosiliceous mineral. The average dye value, per gram kaolinitic mineral present, is 1,925 units, which is greatly in excess of the value for the specimen of kaolinite, but of the same order as that for halloysite, the presence of which has been conjectured on other evidence. Data for the clay fractions of the laterite and the pink earth, however, give an average janus red dye value, per gram of presumed kaolinitic mineral, of 4,650 units, which is considerably higher than that given by any of the minerals examined. This abnormality perhaps may be explained by the supposition that these two layers contain free colloidal silica, though this is scarcely supported by the results of the Truog treatment next to be described.

### *Truog treatment*

As a further test of the nature of the reactive components of the laterite and the pink earth, samples of the materials were examined by the Truog procedure for "removal of free iron oxide, free alumina and colloidal silica" (20), by treatment with sodium sulfide and oxalic acid under alternating alkaline and acidic conditions. The results obtained by chemical analysis of the extracts, and dye values for the residual materials after treatment, are presented in tables 7 and 8. They further demonstrate that the iron oxide component is uncombined, being almost completely removable by the treatment, leaving colorless residues. Some, though not all, of the gibbsitic alumina in the laterite and most of that in the pink earth are dissolved by the treatment. Only a small proportion of the total silica is removed, though apparently not solely the free colloidal silica, and the activity of the aluminosiliceous minerals, as measured by the adsorption of janus red, seems to have been considerably increased by the treatment which possibly enhances their dispersion.

### *Phosphate fixation*

The agricultural importance of free active hydrous iron oxide in soils lies in the ability of this oxide to adsorb and to fix a large amount of phosphate ion (for example, from added soluble manures), which becomes available to plants only under special circumstances (19). In order to examine the phosphate-fixing ability of the Grenada red earth, Beater's method (1) was applied to samples of the four profile materials, as well as to the artificial ferric hydroxide preparation. The results showed that the materials are capable of adsorbing from neutral ammonium phosphate solutions between 6.8 and 14.4 m.e. of phosphate ion per 100 gm., the surface soil adsorbing least, and the laterite layer most, while the laboratory preparation of ferric hydroxide adsorbed 16.8 m.e. The surface soil, though least active, is nevertheless capable of fixing about 0.163 per cent of its weight of $P_2O_5$, or 1,630 p.p.m. $P_2O_5$. From acidic solutions (pH 3.5) each material further adsorbed about 25 per cent additional phosphate.

## CONCLUSIONS

### *Determination and significance of silica-sesquioxide and hydration ratios*

Silica-sesquioxide ratios have been increasingly employed in recent years for assessing the degree of mineral decomposition of rocks and the extent of degradation by leaching of the products of weathering during the subsequent development of soil profiles from the derived parent materials. They have also been applied in attempts to define the terms "laterite" and "lateritic,"

and for distinguishing different types of clay. In view of the fact that certain secondary clay minerals, notably beidellite and montmorillonite, may apparently suffer substitutions of aluminum for silicon and of magnesium and iron for aluminum in the lattice layers (14), great variation in the silica-sesquioxide ratio may be expected, particularly for *sedimentary rocks* that contain specific clay minerals in abundance. Such variation will have no genetic or pedologic significance in these cases. On the other hand, the ratio may have considerable significance in the case of rock products containing primary min-

TABLE 7

*Components removed by Truog treatment*

Values in percentages of original whole material

| MATERIAL* | COMPONENTS REMOVED BY TRUOG TREATMENT | | | ORIGINAL COMPONENTS | | | | |
|---|---|---|---|---|---|---|---|---|
| | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | Free $SiO_2$ | Combined $SiO_2$ | Gibbsitic $Al_2O_3$ | Combined $Al_2O_3$ | Free hydrous $Fe_2O_3$ |
| Laterite | 0.7 | 5.4 | 8.4 | 2.2 | 15.5 | 24.9 | 11.3 | 10.4 |
| Pink earth | 0.8 | 3.0 | 8.0 | 0 | 33.5 | 3.0 | 29.3 | 8.6 |

*100-mesh.

TABLE 8

*Dye values for residual materials after Truog treatment*

Values in dye units per gram of original whole material

| RESIDUAL MATERIAL | BEFORE TREATMENT | | | | | AFTER TREATMENT | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Alizarin* | Diamine sky blue | | | Janus red | Alizarin* | Diamine sky blue | | | Janus red |
| | | Boric | Phosphatic | Difference | | | Boric | Phosphatic | Difference | |
| Laterite | 249 | 390 | 83 | 282 | 704 | 192 | 100 | 0 | 100 | 1304 |
| Pink earth | 30 | 474 | 246 | 254 | 1298 | 20 | 428 | 236 | 113 | 1860 |

*Ignited material.

erals belonging to the kaolin group or consisting of simple mixtures of sesquioxides and silica commonly resulting from the weathering of *igneous rocks* in the humid tropics. Here the determination of the individual free sesquioxides and silica may help considerably in the complete definition of these products.

*Free silica* in the form of quartz may now be determined with considerable accuracy in rock-weathering products by the tri-acid digestion method (7), and in this way it may be distinguished from silica combined in aluminosilicates and ferrosilicates. *Free alumina* likewise may be estimated by the improved alizarin method applied to the *ignited* material, for it is the only component whose capacity for adsorbing alizarin is not greatly diminished by intense heat. Unfortunately, this property applies to amorphous or colloidal alumina as well as to crystalline gibbsite, and it therefore cannot be used for distinguishing between them. Studies of bauxites, bauxitic clays, and laterites

have indicated, however, that free alumina generally occurs naturally as gibbsite, at least in humid tropical rock-weathering products (9). This fact may not preclude the possibility of its occurring in the amorphous form in certain materials such as alkali subsoils, although direct demonstration would be difficult, except by the application of some specific test, and none seems yet to have been elaborated. *Free iron oxide*, in the form of magnetite, where present, may easily be removed and determined directly by weighing.

The advantages of this new departure from the conventional method of expressing the results of chemical analysis of weathering products are revealed by the ratio values given in table 1, which confirm the identity of the halloysitic component of the four kinds of materials comprising the Grenada red earth. Were it not for the fact that quartzose silica, gibbsitic alumina, and magnetic iron oxide could be eliminated by the methods employed, the computation of the probable mineralogical composition of the four layers of the profile would have been impossible. Furthermore, the value of mineralogical determinations, thermal analysis, and measurements of soil constants, as a check on the computations, is clearly demonstrated. Unfortunately, the dye tests as applied to the fresh materials have failed to assess the various iron oxides, although they have indicated their high degree of activity and their ability to combine with considerable amounts of phosphate. The use of similar but more sensitive dyes and of a greater range of authentic reference minerals, together with further improvements in technic, such as the strict standardization of particle size by centrifugal fractionation, would increase the selectivity and precision of the dye tests and is worthy of further study.

### *Classification of parent materials of red soils*

The application of the procedures described and illustrated in this article should materially help in classifying humid tropical and subtropical soils (6), and particularly in further subdividing so-called laterite and lateritic types. In this connection, the writers agree with the view expressed by Marbut (13) that "laterite is a geological material . . . from which more than one type or group of soils may develop"; it may either give rise directly to a red soil (laterite soil), or it may first produce red earth by resilication (as in the case of the Grenada profile), which may then become the parent material of other kinds of red soil, certain of which may be lateritic.

Mineralogically and chemically, the name *laterite* in soil literature has come to mean a rock-product characteristically rich in gibbsite, although other minerals and amorphous substances may also occur in it, such as kaolins (mainly halloysite), hydrous iron oxides, primary and secondary quartz, colloidal silica, manganese oxide, and titania. Alkalies and alkaline earths are typically absent. When iron is especially abundant in the original rock, iron oxide usually accumulates as concretions and crusts or as vesicular slaggy masses, many of which are indurated. The silica-alumina molecular ratio of such a gibbsitic material would be considerably less than 2.0 (the ratio for kaolins), and this fact has given rise to the modern pedologic conception of

laterite as essentially an aluminous material having a silica-alumina ratio less than that of kaolin minerals. Thus, according to Scrivenor (18), "a few analyses have destroyed the value of laterite as a scientific term," because the name originally was given to an Indian building stone having certain characteristic features, such as hardening on exposure, though a low silica-alumina ratio was not among them, as has been proved recently by the examination of authentic specimens (4).

In view of the fact that the mineralogical composition of laterite cannot be determined by conventional chemical analyses, which allow no differentiation between free and combined silica and alumina, recourse must be had to other methods such as those described in this article. By the application of these methods the parent materials of red soils may now be exactly described and defined on the basis of their content of gibbsite, kaolins, and free iron oxides, as well as that of other less essential components. As a beginning, it is tentatively suggested that the parent soil materials might be classified, first, on their gross contents of gibbsite[9] into (a) those that contain much gibbsite (for example, over 20 per cent[10] of their weight), namely, *bauxites* and *laterites*, and (b) those that contain little or none, namely, *primary kaolinic clays*. The materials belonging to each class might then be subdivided on their contents of kaolin minerals, and those belonging to each of these subclasses might be subdivided again on their contents of iron oxide. The proposed provisional nomenclature may be represented schematically as follows:

| GIBBSITE ($Al_2O_3 \cdot 3H_2O$) | KAOLINS ($Al_2O_3 \cdot 2SiO_2 \cdot nH_2O$) | IRON OXIDE ($Fe_2O_3 \cdot nH_2O$) | PROPOSED NAME |
|---|---|---|---|
| *(a) Gibbsitic class (bauxites and laterites)* | | | |
| Much* | Much* | Much* | (i) Kaolinic laterites |
| | | Little | (ii) Bauxitic clays |
| | Little | Much | (iii) Laterites; ferruginous laterites |
| | | Little | (iv) Bauxites (high grade) |
| *(b) Kaolinic class (primary clays)* | | | |
| Little | Much | Much | (i) Ferruginous kaolinic clays |
| | | Little | (ii) Kaolinic clays (white) |
| | Little | Much | (iii) Ferruginous clays |
| | | Little | (iv) (Quartzites, manganese ores, etc.) |

* By "much" is implied more than 20 per cent by weight.

[9] Fermor (3) proposed to differentiate between laterites and lithomargic clays (amorphous kaolinitic or allophanic clays) on the basis of content of "lateritic components," namely, hydrates of alumina, ferric oxide, and titania.

[10] Corresponding to alizarin dye values per gram material after ignition, of 125 units up to 624, the value for pure gibbsite.

The gibbsitic class of rock products presumably includes materials derived from basic and intermediate igneous and metamorphic rocks, whereas the kaolinic class includes china clays, pipe clays, and pot clays mostly derived from acidic rocks such as granite, often stained to different degrees by hydrous iron oxides. This class would also include highly ferruginous products derived from ultra basic rocks (peridotite; serpentine), such as Nipe clay, of Cuba and Puerto Rico. When quartz is plentiful in addition, the descriptive prefix, quartzose, might be employed, and when manganese oxide is abundant, the prefix, manganiferous, might be used.

According to this classification, *laterite* (in the pedologic sense) would be defined as a parent soil material ranging from pure bauxite (or gibbsitic rock; $Al_2O_3$, 65 per cent), to a material containing not less than 20 per cent gibbsite or more than 50 per cent kaolins, with 30 per cent hydrous iron oxides, or with 30 per cent of a mixture of iron oxides, quartz, colloidal silica, and other accessory components. The material representing the lower end of this laterite range would thus have a combined silica content of about 25 per cent, and a total alumina content of about 33 per cent (13 per cent of it being free), giving a silica-alumina molecular ratio below 1.33, which conforms with the standard suggested by Martin and Doyne (15).

With decreasing content of gibbsite and increasing content of kaolins, the material might still be described as *lateritic*, but when the content of gibbsite has diminished to zero, it could no longer be described thus, but should be named *kaolinic clay*, with appropriate prefix denoting the presence of other products. The general terms *red earth* (or *lateritic red loam*) and *red loam* would be equivalent to the terms *ferruginous lateritic clay* and *ferruginous kaolinic clay*, and the term *terra rossa* might be retained for further describing these materials when they accumulate over hard limestones. If, in addition, the type and degree of adsorptive activity of the iron oxide component of ferruginous clays were to be indicated from the results of improved dye tests (for example, by the substitute prefixes *turgitic*, *limonitic*, *hematitic*, etc., these terms having first been standardized), then further definition could be given to the different products of primary rock weathering, and eventually a comprehensive classification of the derived parent soil materials could be elaborated.

In recent years, a tendency has developed for grouping together all red-colored parent soil materials for the purpose of defining soil series, no matter what their origin or mode of formation. Though praiseworthy insofar as it simplifies soil classification, the practice should not be permitted to spread unduly, since it should now be possible to describe and define the parent soil materials in terms other than those of mere coloration and structure. At least in theory, the classification of parent soil materials in all cases should be based on their entire mineralogical composition. In attaining this end, the employment of laboratory methods other than chemical analysis should prove valuable, provided such methods are sufficiently simple and reliable for

routine application. It is hoped that some of the methods described in this article may meet these requirements.

SUMMARY

The main object of the work described in this paper was to investigate various laboratory methods for following the transformations of a basic igneous rock into the parent material of a red soil, and the genesis of soil from it.

The methods used were chemical analysis of whole materials and colloidal clay components, mineralogical examination under the microscope, the construction of mineral dehydration curves, the determination of some physical and chemical soil constants, and dye adsorption tests, including an improved alizarin test for gibbsite and some other tests (diamine sky blue and janus red) for hydrous iron oxides and clay minerals.

These methods were applied to representative materials obtained from a deep red earth profile overlying augite andesite as developed in Grenada, British West Indies, under a humid tropical climate with pronounced wet and dry seasons.

It was found possible by the application of these methods to distinguish and to determine quartzose silica and combined silica, free (gibbsitic) alumina and combined alumina, magnetite, and hydrous iron oxides. The separate determination of the free oxides made it possible to estimate the amounts of secondary aluminosiliceous minerals present in the several materials, and so revealed their identity as kaolins (probably metahalloysite chiefly, with some kaolinite).

The formation of primary laterite from the parent igneous rock and of pink earth and red earth from the laterite is described and discussed. The red earth may be regarded as the parent material of the surface soil, but as no very marked soil development has occurred in this case, the discussion mainly concerns the processes of laterization, gibbsite silication, and the redistribution of iron oxide.

A new theory to account for gibbsite silication as an essential process in the transformation of primary gibbsitic laterite into *secondary* kaolinic red earth is submitted, based on colloid chemical interactions between descending acidic water and alkaline silica solutions comprising ground water the level of which fluctuates within the zone of intermittent saturation coinciding with the pink earth layer. The formation of red earth from pink earth involves no further changes other than the redistribution of hydrous iron oxides, their completed oxidation, partial dehydration, and equalization.

In view of the great agricultural importance of the phosphate relations of ferruginous soils, the adsorptive properties of the hydrous iron oxide components of the different profile materials were examined by measuring phosphate uptake, as well as that of certain dyes, and comparing the results with those obtained with authentic specimens of different iron oxide minerals, clay minerals, and laboratory preparations of hydroxides.

It is concluded that the significance and value of silica-sesquioxide ratios

may be considerable in the case of igneous rock-weathering products and soils, though doubtful in the case of sedimentary rock products containing secondary expanding clay minerals exhibiting ionic replacements in the lattice layers. The separate determination of free quartzose silica and of free gibbsitic alumina facilitates the interpretation of silica-alumina ratios in primary products of igneous rock weathering and further demonstrates the kaolinic nature of the aluminosiliceous component of the four materials comprising the red earth soil profile.

The classification of the parent materials of red soils is discussed in the light of these findings, and a provisional scheme and a nomenclature are submitted, based on determinations of their contents of gibbsite, kaolins, and iron oxide, and of less essential components, such as quartz, manganese oxide, and titania. The terms *laterite*, *lateritic*, and *kaolinic* are defined on this basis, and it is submitted that a more satisfactory description and definition of parent materials of red soils may be attained by these means than by field observations alone, and this should permit greater precision in identifying soil series.

## APPENDIX

### *Dye adsorption tests for gibbsite, hydrous iron oxides, and clay minerals*

*The alizarin method for estimating gibbsitic alumina.* The details of the modified method are as follows:

Two 1-gm. portions of air-dried material ground to pass a 100-mesh sieve, contained in silica weighing bottles, are ignited in a thermo-regulated electric muffle furnace at 750°C. for 15 minutes. After cooling, the ignited material is transferred to large boiling tubes, one containing 20 cc. of a 0.5 per cent solution of alizarin red S in boric alcohol, the other containing 20 cc. of boric alcohol alone. Both tubes are fitted with Hopkins-Cole reflux condensers and heated in a boiling-water bath for 10 minutes. The supernatant liquid in each tube is decanted into a 30-cc. silica Gooch crucible containing a pad of filter-paper pulp. In each case, the soil material in the tube is treated once with 20 cc. boric alcohol, boiled, and the whole transferred to the crucible and filtered by suction. Excess of dyestuff is next removed in one case by washing with several portions of boiling water until the filtrate is quite colorless. The duplicate ("blank") treatment is carried out in exactly the same manner, the material being given the same number of washings.

The stained portion is now "stripped" by treatment with boiling saturated aqueous sodium oxalate-oxalic acid solution at pH 3.8. The paper pad and stained material are returned to the boiling tube, adhering particles being washed off the crucible with oxalate solution. The volume is made up to about 30 cc., and the mixture is boiled, settled, and decanted into the Gooch crucible containing a fresh pad of paper pulp. The washing is repeated several times with small quantities of oxalate solution, and the volume is finally brought to a definite amount for the purpose of colorimetric comparison with a standard.

Suitable alizarin standards may be prepared by diluting 0.25 to 2.0 cc. of a stock 0.5 per cent aqueous solution of alizarin red S (the amount taken depending on the color of the "unknown") with an appropriate volume of "blank" oxalate extract of the unstained material; this corrects for color caused by iron and organic compounds soluble in the oxalate solution. One cubic centimeter of a 0.5 per cent dyestuff solution is taken as containing 100 dye units; thus, if both extract and standard are diluted to the same volume, and the standard contains 1.0 cc. of dyestuff solution, then, if the colors are exactly matched, the material has fixed 100 dye units per gram. Under such conditions, *ignited* gibbsite adsorbs

1,000 dye units per gram $Al_2O_3$. Hence the percentage content of gibbsitic alumina in a sample under examination is obtained by multiplying its dye volume in units by 0.1.

The alizarin value of fresh *unignited* material is determined in exactly the same manner, the "blank" correction being applied in every case.

*Preparation of Reagents.* (a) *Alizarin solution.* Dissolve 5.0 gm. alizarin red S in 100 cc. water; complete the solution with 80 per cent alcohol saturated with boric acid (pH 3.2), and make up to 1 liter with boric alcohol. (b) *Oxalate solution.* Dissolve 42 gm. oxalic acid in 800 cc. water, add sufficient 40 per cent sodium hydroxide solution to give a reaction of pH 3.8, and make up to 1 liter with water. Filter off before use any sodium oxalate that may crystallize. (c) *Boric alcohol.* Dissolve 33 gm. boric acid in 1 liter of 80 per cent alcohol and filter off undissolved excess. Reaction, pH 3.2.

*The diamine sky blue and the janus red tests.* (a) *In borate solution:* Two 1-gm. portions of 100-mesh air-dried material are treated, one with 20 cc. dyestuff solution prepared by dissolving 0.2 gm. in a small volume of water and diluting to 100 cc. with 80 per cent alcohol saturated with boric acid, the other with 20 cc. boric-alcohol alone. The liquids are then decanted into Gooch crucibles containing disks of filter paper covered with glass dust, the subsequent treatments being the same as those in the alizarin test. "Stripping" is effected by means of alcoholic sodium hydroxide solution, prepared by mixing equal volumes of normal soda and 90 per cent alcohol. (Generally, the stripped material still retains some dye which cannot be removed except by drastic treatment.) Colorimetric comparison is conducted against a standard prepared by diluting an appropriate volume of the original 0.2 per cent dyestuff solution with "blank" extract, exactly as in the alizarin test. (b) *In phosphate solution:* The procedure is the same, except that a solution of the dyestuff in alcohol containing phosphate is employed instead of boric-alcohol. This is prepared by dissolving 0.2 gm. dyestuff in a small quantity of water and adding to it 10 cc. of an aqueous solution containing 2 gm. potassium dihydrogen phosphate ($KH_2PO_4$). The mixture is then diluted to 100 cc. with alcohol and water in such proportion that no precipitate forms. The reaction of the final reagent is about pH 5. A "blank" extract is also prepared for diluting the standard for colorimetric comparison.

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# THE EFFECT OF FREE IRON OXIDE REMOVAL ON SOME PROPERTIES OF SOIL COLLOIDS[1]

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In a previous contribution (9) some preliminary data were presented on the role of the free iron oxides in the phosphate-ion adsorption by soil colloids. It was pointed out that the removal of the free iron oxides from the colloids decreased the adsorption of phosphate ions. The phosphated residue, as a result of the decreased adsorption, possessed a smaller cation exchange capacity.

The purpose of the present investigation is to present further evidence to show that the treatment used for the removal of the free oxides of Fe, Al, and Si partially destroys the basoid properties of the colloids. The changes in the colloidal nature of the deferrated colloids have been approached essentially from the belief in the amphoteric behavior of the soil complexes (7). The data presented deal with ion adsorption, exchange capacity values, ultimate pH, and electrodialytic behavior of untreated and deferrated colloids.

For this study an attempt was made to obtain a series of colloids which would contain variable amounts of free iron oxides. Colloids were extracted from the following soil types by the method described previously (9):

*Cecil clay subsoil:* A new sample extracted from the 10–28-inch depth of a profile near Statesville, N. C.

*Colts Neck loam:* The A horizon of a virgin profile approximately 1½ miles southeast of Red Bank, N. J.

*Penn silt loam:* The middle of the B horizon of a virgin profile collected from a road cut about 2 miles south of Franklin Park on the road to Griggstown, N. J.

*Sassafras loam:* A new sample of the lower B horizon of a profile on the Wolpert Farm of the New Jersey Agricultural Experiment Station, New Brunswick.

## EXPERIMENTAL METHODS

After extraction from the soils, the colloids were electrodialyzed free of exchangeable bases (8), and their chemical composition was established by the methods used for soil analysis. The composition is expressed in terms of samples dried to constant weight at 30°C.

The method of Drosdoff and Truog (4) was used to obtain the total free iron oxide content of the colloids. To obtain variations in the amounts of free iron

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oxide remaining in the colloid residue, the technic of the method was modified as follows: The series designated as treatment "A" in the following discussions were not digested with 2.5 per cent $Na_2CO_3$ solution but were rendered partially Na-saturated by washing twice with 50 cc. of *N* NaCl solution before being saturated with $H_2S$. The "B" series received the full treatment. After the removal of the oxides and free sulfur, the colloids of the two series were again partially Na-saturated by the previous treatment before being electrodialyzed. This procedure was followed to aid in identifying the end point of electrodialysis as previously pointed out (8).

The anion adsorption experiments were generally performed at pH values that ranged from 2 to 6 and at concentration ranges of from 20–40 m.e. of anions per 10 gm. of colloid. The variations in pH ranges for the chloride system were obtained by mixing normal solutions of HCl and $NH_4OH$. With the phosphated series, varying pH values resulted when 0.5 *N* solutions of $(NH_4)_2HPO_4$ and $H_3PO_4$ were mixed. The quantity of chloride ion adsorbed was determined gravimetrically by precipitating and weighing the chloride as AgCl from an aliquot of the supernatant liquid of each system after adsorption. The usual volumetric procedure was used for determining the amount of phosphate ion adsorbed from an aliquot of the supernatant liquid after the removal of displaced silica by the usual method.

The cation exchange capacities of the original and deferrated electrodialyzed colloids were determined by the barium acetate method (8). For the determination of the exchange capacity of the phosphated colloids, the samples, after ion adsorption, were transferred to a filter with 100 cc. of a normal $NH_4Ac$ solution and then leached to 250 cc. with this solution to remove excess soluble phosphates prior to treatment of the samples with barium acetate solution.

The cation-saturated colloids were prepared by leaching 25 gm. of the H-colloids with 6 liters of normal neutral acetate solutions of the cations and then washing with 50 cc. of normal chloride solutions of the cations. The excess electrolyte was removed by washing with distilled water until the samples were free of chloride ions. The exchangeable cation content of the prepared colloids was determined by displacing the cations from a 2-gm. sample with a solution of normal neutral $NH_4Ac$ and analyzing the filtrate for Ca, Ba, or Mg by the usual methods. For studying the release of cations by electrodialysis, a 10-gm. sample was placed in a Mattson cell and electrodialyzed at a constant amperage of 0.4 for 24 hours in the case of the Ca- and Ba-colloids and for 48 hours in the case of Mg-colloids. Silica was removed from the catholyte prior to determining the cations by the usual methods. The residual cations not released by electrodialysis from the colloids were displaced by 500 cc. of normal neutral $NH_4Ac$ and determined as described above.

The electrodialyzed $SiO_2$ and $R_2O_3$ in the catholyte were determined by stabilizing the silica as $SiO_2$ and precipitating the $R_2O_3$ in the filtrate with $NH_4OH$. Iron was determined by reduction with stannous chloride and titration with 0.1 *N* $KMnO_4$.

The neutralization curves of the untreated and deferrated colloids were obtained by the method of Anderson and Byers (2).

The pH values of all samples were obtained by the use of a glass electrode on colloid suspensions.

### EFFECT OF THE DROSDOFF-TRUOG METHOD ON CERTAIN COLLOID PROPERTIES

The chemical composition of each of the colloid fractions extracted from the soils is presented in table 1. The silica-sesquioxide ratios of the colloids vary from 1.05 for the Cecil to 3.90 for the Sassafras. The composition of the Cecil and that of the Sassafras are identical with those reported previously for these colloids (9). The silica-sesquioxide ratios are somewhat higher because of the correction for the titanium which has been determined. The Colts Neck colloid isolated from the A horizon of the profile described previously is lower both in total $Fe_2O_3$ and $Al_2O_3$ than the colloid isolated from the B horizon of this profile. The free iron oxide content of the A horizon is, however, higher than that of the B.

The data presented in table 2 were obtained, as has been pointed out, by varying the technic of the Drosdoff-Truog method (4). This procedure was used rather than the newer modification (10), since the latter method resulted in a more intense degradation of the complex.

The differences between the free iron oxide contents of the colloids as determined by treatments A and B (table 2) may be explained in part by the following observations: During the digestion of the colloids with sodium carbonate solution, it was noted that mobilization of organic matter was more intense from the colloids that show differences in the free iron oxide content greater than 2 per cent by the two technics than from the others which did not show this variation. It is possible that humic acid gels protect the free iron oxides from reacting with the $H_2S$. If these gels are removed by digestion with alkali carbonates, however, the oxides do react. This preliminary digestion with the alkali carbonate also enhances the breakdown in acid of the silicate minerals. This fact is confirmed by the data in tables 2 and 3, which show a definite increase in silicate solubility by method B. The data in table 2 indicate only slight effects of treatment A on the cation exchange capacity of the colloids, whereas treatment B markedly affected the exchange power of the Colts Neck colloid. In the light of these findings, therefore, large batches of deferrated colloids were prepared by treatment A for later work. It should also be pointed out that if the organic matter had been removed from the colloid prior to determining the free iron oxide content, probably only slight effects would have been noted upon the exchange capacity. It is the author's belief, however, that soil colloids and their properties should be studied on samples as isolated from the soils, since the role of the organic matter fraction in colloids is equally as important as the role of the inorganic fraction.

The loss in weight of the various colloids as affected by the two treatments

TABLE 1

*Chemical composition of soil colloids*

| COLLOID | $\frac{SiO_2}{R_2O_3}$ | $SiO_2$ | $TiO_2$ | $P_2O_5$ | $Fe_2O_3$ | $Al_2O_3$ | MnO | CaO | MgO | $K_2O$ | C | WATER (105°C.) | IGNITION LOSS | CATION EXCHANGE CAPACITY (pH 7.0) | $pH_H$ ($H_2O$) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | *per cent* | *per cent* | *per cent* | *per cent* | *per cent* | *per cent* | *per cent* | *per cent* | *per cent* | *per cent* | *per cent* | *per cent* | *m.e./gm.* | |
| Cecil | 1.05 | 34.48 | 0.68 | 0.05 | 14.34 | 45.76 | 0.19 | 0.04 | 0.58 | 0.42 | 0.48 | 2.23 | 4.00 | 0.107 | 5.30 |
| Colts Neck | 1.11 | 26.49 | 0.61 | 0.88 | 32.71 | 19.25 | 0.15 | 0.14 | 0.78 | 1.50 | 1.78 | 5.19 | 12.18 | 0.288 | 4.50 |
| Penn | 2.26 | 45.05 | 0.35 | 0.06 | 8.54 | 28.34 | 0.12 | 0.14 | 1.98 | 3.57 | 0.41 | 4.04 | 7.76 | 0.225 | 4.60 |
| Sassafras | 3.90 | 53.02 | 0.76 | 0.36 | 15.69 | 15.89 | 0.16 | 0.36 | 0.79 | 0.42 | 0.79 | 3.25 | 6.49 | 0.206 | 5.00 |

TABLE 2

*Effect of free iron oxide removal upon cation exchange capacity and ultimate pH*

| COLLOID | TREATMENT A* | | | TREATMENT B† | | | CATION EXCHANGE CAPACITY | | | $pH_H$ ($H_2O$) | | | $pH_H$ (KCl) | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Residue from 1-gm. sample | Per cent of total $SiO_2$ dissolved | Free $Fe_2O_3$ | Residue from 1-gm. sample | Per cent of total $SiO_2$ dissolved | Free $Fe_2O_3$ | Original colloid | Residue from treatment A | Residue from treatment B | Original colloid | Residue from treatment A | Residue from treatment B | Original colloid | Residue from treatment A | Residue from treatment B |
| | *gm.* | | *per cent* | *gm.* | | *per cent* | *m.e./gm.* | *m.e./gm.* | *m.e./gm.* | | | | | | |
| Cecil | 0.850 | 1.23 | 10.98 | 0.780 | 2.26 | 12.32 | 0.107 | 0.094 | 0.106 | 5.30 | 4.45 | .... | 5.10 | 4.45 | .... |
| Colts Neck | 0.667 | 3.39 | 19.58 | 0.484 | 9.47 | 28.27 | 0.288 | 0.302 | 0.205 | 4.50 | 4.30 | 4.10 | 4.20 | 4.20 | 4.00 |
| Penn | 0.896 | 1.24 | 3.58 | 0.827 | 4.75 | 4.96 | 0.225 | 0.237 | 0.202 | 4.60 | 4.45 | 4.10 | 4.25 | 4.30 | 4.00 |
| Sassafras | 0.837 | 1.76 | 6.13 | 0.772 | 4.09 | 7.58 | 0.206 | 0.204 | 0.208 | 5.00 | 4.35 | .... | 4.90 | 4.30 | .... |

* No preliminary digestion with 2.5 per cent $Na_2CO_3$.

† Preliminary digestion with 2.5 per cent $Na_2CO_3$.

is high, but it is surprising to note that the only colloid affected to any marked extent with respect to variations in exchange powers was the Colts Neck with treatment B. These results, in general, confirm previous data (9), namely, that only those colloids which possess acidoid fractions which mobilize at high pH values show reductions in exchange powers by the Drosdoff-Truog method. The greatest loss in weight due to treatment B, found with the Colts Neck colloid, represented nearly 50 per cent of the original weight based on oven-dried samples. Of this loss, approximately 35 per cent (table 3) represented solubility of the inorganic fraction. The residual loss of nearly 15 per cent is probably due to a loss of water of hydration from the destruction of the aluminum and iron silicates and solubility of organic matter. Losses from the Cecil, Penn, and Sassafras colloids were smaller than those from the Colts

TABLE 3

*Composition of acid-soluble and electrodialyzable fractions from the Colts Neck and Penn colloids*

Results in grams per 100 gm.

| CONSTITUENT | ACID-SOLUBLE FRACTION ($H_2S$ AND HCl) | | ELECTRODIALYZABLE FRACTION FROM RESIDUE | | ELECTRODIALYZABLE FRACTION FROM ORIGINAL COLLOID | |
|---|---|---|---|---|---|---|
| | Colts Neck | Penn | Colts Neck | Penn | Colts Neck | Penn |
| $SiO_2$ | 2.032 | 1.251 | 0.080 | 0.051 | 0.038 | 0.010 |
| $Fe_2O_3$ | 28.270 | 4.960 | 0.029 | 0.010 | 0.012 | 0.006 |
| $Al_2O_3$ | 5.208 | 2.381 | 0.085 | 0.029 | 0.025 | 0.015 |
| CaO | 0.038 | 0.024 | 0.008 | 0.007 | Trace | Trace |
| MgO | 0.113 | 0.198 | 0.016 | 0.011 | Trace | Trace |
| Total | 35.661 | 8.814 | 0.218 | 0.108 | 0.075 | 0.031 |
| $SiO_2/Al_2O_3$ | 0.67 | 0.91 | 1.60 | 3.03 | 2.16 | 1.14 |

Neck and ranged from 18 to 23 per cent. The smaller losses from these colloids are due mainly to their lower content of free iron oxides.

It is generally admitted that the free oxides of Fe, Al, and Si possess little or no significant cation exchange powers (9); therefore, the removal of these constituents should not alter the exchange capacity of colloids. During the acid digestion of the black iron sulfides, however, fairly high amounts of Al, Si, Ca, and Mg are brought into solution (table 3) without affecting the exchange powers of certain colloids. Since the composition of the acid-soluble fraction for the colloids by treatment A is not available, only general statements can be made in regard to the solubility of the former constituents by this treatment. It is safe to assume that the effects of treatment A are less intense than those of treatment B (table 2), but still considerable quantities of silica and alumina are brought into solution. It would not be reasonable to assume that these constituents exist as free oxides in the colloids in such large quantities, and the general data do not indicate this. The only conclusion one can draw is

that a degradation of the complex results. In the light of these facts, what inferences can be drawn in regard to the acid solubility of colloidal material such that only slight effects are noted on the exchange capacity? It is possible that a differential solubility of aluminum silicates results and that these silicates are of low silica-sesquioxide ratio (0.5–1.0) and of low exchange capacity.

The pH values of the electrodialyzed colloids in water and in *N* KCl before and after the removal of the free iron oxides by both methods are presented in table 2. The ultimate pH values in water and in KCl solution of the untreated colloids bear a definite relation to the cation exchange capacity (7, 9). Because of exchange reactions, the values in KCl solutions are lower than those in water.

The electrodialyzed residues of treatment A with about the same exchange capacity as the untreated colloids have lower pH values in both water and KCl solutions than do the untreated colloids. The pH values are about the same for all the deferrated colloids, and no relation exists between the ultimate pH and the exchange capacity. The complete removal of the free oxides from the Colts Neck and Penn colloids has further reduced the ultimate pH by about 0.1 to 0.3 unit. The significance of the lowering of the ultimate pH of the colloids by the removal of the free oxides was at first questioned. This was due, in part, to the belief that the $CS_2$ used to extract the free sulfur may have been incompletely removed prior to and during electrodialysis. Since $CS_2$ is acid, the incomplete removal from the colloids might have lowered the ultimate pH. To test this point, the treatment for the removal of the free oxides was repeated, and simultaneously untreated colloids were subjected to similar washings with $CS_2$ and alcohol. The samples after washing, but in the moist condition, were placed on a steam bath, and the deferrated samples after this treatment were electrodialyzed as usual. Determinations of pH on the washed untreated samples and deferrated colloids yielded the same pH values as those recorded in table 2. From this behavior it was concluded that the effect of free oxide removal on reductions in the ultimate pH of the residues was correct.

To account for this behavior, one must conclude that the reduction in ultimate pH must be due, in part, to the $H_2S$, dilute acid treatment, and electrodialysis. The colloids, whether subjected to treatments A or B, lost a part of the nonexchangeable cations by acid solubility and hydrolysis. This is definitely indicated by the data in table 3 for treatment B. The former effects are less intense for treatment A than for treatment B. It should be noted from the data in table 3 that whereas the Colts Neck and Penn colloids lost 0.151 and 0.222 gm. per 100 of CaO plus MgO by solution and electrodialysis, the untreated colloids when rendered Na-saturated and electrodialyzed, yielded only traces of CaO and MgO. When hydrolyzed for 1 week in distilled water, however, the original colloids showed increases in ultimate pH as follows: Cecil, 0.3 unit; Colts Neck, 0.8 unit; Penn and Sassafras colloids, 0.4 unit, whereas the deferrated residues of treatment A showed only very slight elevations and those of treatment B showed none. Whether the loss of the part of

the nonexchangeable cations made soluble by the acid treatment and by electrodialysis can account for this variation in ultimate pH is uncertain. It is also possible that the removal of the gel envelopes by the free iron oxide treatment activates some of the acidoid fractions, and as a consequence an increased dissociation of H ions results (3).

The data presented in table 3 are in some respects similar to those reported by Truog and associates (10). The effects of the free iron oxide removal upon the release of aluminum and silica have been noted by these investigators. The effects of the treatment upon the release of a part of the nonexchangeable cations and the electrodialytic behavior of the deferrated colloids, however were not studied by Truog. The quantity of aluminum, silica, and calcium rendered soluble by the treatment is apparently independent of the composition of the colloid. This is indicated by the fact that the Colts Neck colloid with a silica-sesquioxide ratio of 1.11 lost more of these constituents as a result of the treatment than did the Penn colloid with a silica-sesquioxide ratio of 2.26. On the assumption that the silica and aluminum brought into solution represent the degradation of aluminosilicates of a definite composition, the silica-alumina ratios of the soluble fractions of the two colloids are fairly uniform, being 0.67 for the Colts Neck and 0.91 for the Penn colloid.

A comparison of the composition of the catholyte sediments from the original and deferrated residues brings out several interesting facts. The release of silica and the sesquioxides from the deferrated residues is greater than that from the untreated colloids. This may be due, in part, to the effect of the high pH on hydrolyzing silicate bonds in the clay lattices. If a hydrolysis of this nature occurred, however, a corresponding increase in exchange capacity would have resulted, and therefore the new complex would yield more silica and sesquioxides during electrodialysis (9). Since this increase was not noted, the alternative conclusion is that the removal of the oxide gel layers from around the crystalline nucleus enhances the degradation of the deferrated colloids. It is also surprising to note that the transport of iron during electrodialysis is greater with the deferrated residues of the Colts Neck and Penn colloids than with the original colloids even though 6 to 28 per cent of the total iron has been removed by the treatment. An explanation for this is that the free iron oxides in the original colloids are not mobilized during electrodialysis (9). Not all colloids, however, behave as do the Colts Neck and Penn upon partial deferration. In table 4 are presented data with reference to the release of silica, iron, and aluminum during the electrodialysis of Mg-saturated colloids. Although the release of silica and of total $R_2O_3$ is always greater with the deferrated than with the original colloids, the composition of the $R_2O_3$ precipitate varies. In some cases, as with the Colts Neck and Penn colloids, the release of iron from the deferrated colloids is greater than that from the original colloids, whereas with the Cecil and Sassafras, the release is greater from the untreated colloids. With all the colloids, the release

TABLE 4

*Release of $SiO_2$ and $R_2O_3$ from original and from deferrated Mg-saturated colloids during electrodialysis*

Results in milligrams per 100 gm.

| COLLOID | TREATMENT | $SiO_2$ | $R_2O_3$ | $Fe_2O_3$ | $Al_2O_3$ | $\frac{SiO_2}{Al_2O_3}$ COLLOID | CATHOLYTE SEDIMENT $\frac{SiO_2}{Al_2O_3}$ |
|---|---|---|---|---|---|---|---|
| Cecil | None | 30.0 | 72.0 | 22.0 | 50.0 | 1.26* | 1.02 |
| | Deferrated | 72.0 | 140.0 | 8.0 | 132.0 | ..... | 0.93 |
| Colts Neck | None | 48.0 | 128.0 | 55.2 | 72.4 | 2.34* | 1.11 |
| | Deferrated | 204.0 | 360.0 | 103.4 | 256.6 | ..... | 1.35 |
| Penn | None | 80.0 | 96.0 | 22.0 | 74.0 | 2.69* | 1.41 |
| | Deferrated | 92.0 | 128.0 | 32.0 | 96.0 | ..... | 1.62 |
| Sassafras | None | 66.0 | 128.0 | 27.0 | 101.0 | 5.70* | 1.00 |
| | Deferrated | 72.0 | 180.0 | 22.0 | 158.0 | ..... | 0.77 |

* Of untreated colloids.

TABLE 5

*Adsorption of chloride ion by untreated and by deferrated colloids*

| COLLOID | $Cl^-$ ADDED AS HCl + $NH_4OH$ | FREE IRON OXIDE CONTENT | | pH OF SUPERNATANT LIQUID | | $Cl^-$ ADSORBED | |
|---|---|---|---|---|---|---|---|
| | | Original | Deferrated A | Original | Deferrated A | Original | Deferrated A |
| | *m.e./10 gm.* | *per cent* | *per cent* | | | *m.e./10 gm.* | *m.e./10 gm.* |
| Cecil | 20.00 | ..... | .... | 3.70 | 3.70 | 0.320 | 0.000 |
| | 20.00 | 12.32 | 1.44 | 2.00 | 2.10 | 0.480 | 0.000 |
| | 20.00 | ..... | .... | 1.80 | 1.80 | 0.520 | 0.020 |
| Colts Neck | 20.00 | ..... | .... | 3.90 | 3.50 | 0.040 | 0.000 |
| | 20.00 | 25.27 | 8.69 | 2.20 | 2.00 | 0.070 | 0.003 |
| | 20.00 | ..... | .... | 1.80 | 1.80 | 0.320 | 0.010 |
| Penn | 20.00 | ..... | .... | 3.30 | 3.30 | 0.270 | 0.000 |
| | 20.00 | 4.96 | 0.58 | 1.90 | 1.90 | 0.380 | 0.000 |
| | 20.00 | ..... | .... | 1.60 | 1.60 | 0.480 | 0.070 |
| Sassafras | 20.00 | ..... | .... | 4.00 | 4.00 | 0.070 | 0.000 |
| | 20.00 | 7.58 | 1.45 | 2.25 | 2.10 | 0.380 | 0.000 |
| | 20.00 | ..... | .... | 1.75 | 1.75 | 0.500 | 0.027 |

of aluminum is always greater from the deferrated residues. The silica-aluminum ratio of the catholyte sediment is always less than that of the original colloid. This behavior has been discussed elsewhere (9).

## CHANGES IN ANION ADSORPTIVE ABILITY OF SOIL COLLOIDS RESULTING FROM REMOVAL OF FREE IRON OXIDES

The essential changes in the properties of soil colloids that might be expected as a result of the removal of the free iron oxides are reductions in the ability of the clays to adsorb anions, variations in the mechanism of the adsorption, and possible changes in neutralization curves at high pH values. The data presented here deal with the adsorption of both chloride and phosphate ions by the original and by the deferrated colloids. The nature and the mechanisms involved in the adsorption of phosphate ions by the completely deferrated residues are also discussed.

The data presented in table 5 are concerned with the adsorption of chlorides by the various colloids. If the adsorption of this ion is based on the order of activity manifested by the ultimate pH of the colloids, the order should be Cecil, Sassafras, Penn, and Colts Neck. At the lowest pH for the various systems of the original colloids, this order is followed, for the Cecil adsorbs 0.52 m.e., the Sassafras 0.50 m.e., the Penn 0.48 m.e., and the Colts Neck 0.38 m.e. per 10 gm. It is also to be noted that the adsorption increases with decreasing pH (7, 9). The partial removal of the free iron oxides from the colloids has decreased the adsorption of the chloride ion. This significant fact points toward a definite role of the free iron oxides in the adsorption of chloride ions. Since the hydrated iron oxides are responsible for phosphate adsorption, as pointed out by Ford (5), the possibility that chloride ion adsorption may be due to these oxides is suggested here.

The adsorption of phosphate ions by the differently treated colloids is presented in table 6. Certain general conclusions with respect to the adsorption of these anions by the colloids may be deduced from the data. These general facts are that phosphate ion adsorption increases both with increasing anion concentration and with decreasing pH. These points were discussed previously (9). The dependence of the magnitude of adsorption by the colloids upon the silica-sesquioxide ratio, free iron oxide content, and displaced silicates is an outstanding point in table 5. In addition, several significant new facts are apparent.

The phosphate ion adsorption decreases with the completeness of removal of the free iron oxides from the soil colloids, but even with the completely deferrated residue, the adsorption, unlike that of chloride, is rather great. Since it has been pointed out previously (9) that the retention of phosphate ions by the hydrated iron oxides occurring in the colloids is responsible, in part, for the increases in cation exchange capacity, the partial removal of these minerals markedly reduces the cation exchange capacity of the phosphated residues. With the Colts Neck and Sassafras colloids of the treatment B series, phosphate ion adsorption was not associated with increases in cation exchange capacity even though the ion adsorption of the residues amounted to 25 to 50 per cent of the total adsorbed by the original colloids. With the Cecil

TABLE 6

*Adsorption of phosphate ions by untreated and by deferrated colloids*

| COLLOID | FREE $Fe_2O_3$ CONTENT | | | $PO_4^{=}$ ADDED AS $H_3PO_4$ + $NH_4OH$ | pH OF SUPERNATANT LIQUID | | | $PO_4^{=}$ ADSORBED | | | CATION EXCHANGE CAPACITY | | | SILICA IN SUPERNATANT LIQUID | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Original | Treatment A | Treatment B | | Original | Treatment A | Treatment B | Original | Treatment A | Treatment B | Original | Treatment A | Treatment B | Original | Treatment A | Treatment B |
| | *per cent* | *per cent* | *per cent* | *m.e./10 gm.* | | | | *m.e./10 gm.* | *m.e./10 gm.* | *m.e./10 gm.* | *m.e./10 gm.* | *m.e./10 gm.* | *m.e./10 gm.* | *m.e./10 gm.* | *m.e./10 gm.* | *m.e./10 gm.* |
| Cecil | 12.32 | 1.44 | 0.00 | 0.00 | 5.30* | 4.30* | .... | .... | .... | .... | 1.07 | 0.94 | 1.06 | .... | .... | .... |
| | | | | 25.00 | 5.60 | 4.50 | .... | 4.62 | 1.29 | .... | 1.66 | 1.45 | .... | 3.73 | 3.72 | .... |
| | | | | 30.00 | 3.55 | 3.20 | 3.40 | 4.71 | 2.17 | 0.86 | 2.02 | 1.62 | 1.25 | 4.24 | 5.76 | 5.68 |
| | | | | 40.00 | 3.00 | 2.85 | 2.85 | 7.41 | 4.80 | 2.54 | 2.30 | 1.71 | 1.48 | 5.76 | 5.60 | 8.10 |
| Colts Neck | 28.27 | 8.69 | 0.00 | 0.00 | 4.50* | 4.30* | .... | .... | .... | .... | 2.88 | 3.02 | 2.05 | .... | .... | .... |
| | | | | 25.00 | 6.02 | 5.90 | .... | 5.48 | 3.09 | .... | 4.96 | 3.21 | .... | 6.73 | 4.80 | .... |
| | | | | 30.00 | 4.38 | 3.68 | 3.69 | 7.03 | 3.67 | 3.25 | 5.24 | 3.55 | 2.00 | 9.40 | 5.60 | 5.60 |
| | | | | 40.00 | 3.20 | 2.92 | 2.70 | 9.26 | 5.45 | 5.06 | 5.67 | 3.78 | 2.05 | 9.60 | 7.36 | 7.12 |
| Penn | 4.96 | 0.58 | 0.00 | 0.00 | 4.60* | 4.45* | .... | .... | .... | .... | 2.25 | 2.37 | 2.02 | .... | .... | .... |
| | | | | 25.00 | 4.80 | 5.00 | .... | 5.19 | 2.46 | .... | 3.24 | 2.89 | .... | 5.12 | 4.52 | .... |
| | | | | 30.00 | 3.62 | 3.58 | 3.28 | 5.37 | 3.55 | 2.30 | 3.75 | 3.10 | 2.48 | 7.36 | 6.29 | 8.00 |
| | | | | 40.00 | 3.05 | 2.85 | 2.71 | 6.23 | 4.80 | 2.69 | 3.92 | 3.36 | 2.56 | 8.56 | 7.68 | 8.00 |
| Sassafras | 7.58 | 1.45 | 0.00 | 0.00 | 5.00* | 4.35* | .... | .... | .... | .... | 2.06 | 2.04 | 2.08 | .... | .... | .... |
| | | | | 25.00 | 5.73 | 4.70 | .... | 3.33 | 2.61 | .... | 2.84 | 2.52 | .... | 6.00 | 7.26 | .... |
| | | | | 30.00 | 3.88 | 3.35 | 3.38 | 5.49 | 2.63 | 2.03 | 3.38 | 2.72 | 2.07 | 6.96 | 7.68 | 9.12 |
| | | | | 40.00 | 3.00 | 2.80 | 2.70 | 8.84 | 4.28 | 2.30 | 3.87 | 2.86 | 2.06 | 7.04 | 8.16 | 8.82 |

* 1:10 suspensions.

and Penn colloids only slight increases were found in cation exchange capacity of the treatment B residues after phosphate ion adsorption.

Two reasons might be advanced for the adsorption by the colloid residues of the B series without appreciable increases in cation exchange capacity. The adsorbed phosphate ions might be so weakly retained by the residues that the preliminary washing with *N* $NH_4Ac$ and $BaAc_2$ completely displaced the adsorbed ions. To clarify this point, samples of the original and of the residues from the B treatments of the Colts Neck and Penn colloids were allowed to adsorb phosphate ions from a mixture of phosphoric acid and ammonium phosphate solutions. The cation exchange capacities of the treated colloids were determined by the procedure outlined in the experimental methods. The total phosphate content of the original and phosphated colloids was

TABLE 7

*Adsorption of phosphate ions by untreated and by deferrated Colts Neck and Penn colloids in relation to changes in exchange capacity*

| COLLOID | $PO_4^{\equiv}$ ADDED AS $H_3PO_4 + (NH_4)_2HPO_4$ | pH OF SUPERNATANT LIQUID | $PO_4^{\equiv}$ RETAINED | CATION EXCHANGE CAPACITY | INCREASE IN CATION EXCHANGE CAPACITY | SILICA IN SUPERNATANT LIQUID |
|---|---|---|---|---|---|---|
| | *m.e./12 gm.* | | *m.e./12 gm.* | *m.e./12 gm.* | *m.e./12 gm.* | *m.e./12 gm.* |
| Colts Neck—"O" | 0.0 | .... | .... | 3.43 | .... | .... |
| | 25.0 | 5.65 | 4.75 | 5.82 | 2.39 | 1.19 |
| Colts Neck—"B" | 0.0 | .... | .... | 2.46 | .... | .... |
| | 25.0 | 5.65 | 1.62 | 2.50 | 0.04 | 2.43 |
| Penn—"O" | 0.0 | .... | .... | 2.75 | .... | .... |
| | 25.0 | 4.55 | 2.76 | 4.41 | 1.66 | 1.39 |
| Penn—"B" | 0.0 | .... | .... | 2.42 | .... | .... |
| | 25.0 | 4.45 | 1.16 | 2.68 | 0.26 | 2.00 |

determined on the samples used for exchange capacity determinations after the displacement of the adsorbed Ba by 0.05 *N* HCl. These data, presented in table 7, definitely indicate that the strength of the retention of the adsorbed phosphate ions by the deferrated residues and the original colloids is great enough to prevent displacement by salt solutions. The data in table 7 also confirm the facts found from the previous table.

Since it has previously been noted (9) that the adsorption of phosphate ions by electrodialyzed soil colloids may be due in part to the displacement of silicate ions from the lattice by phosphate ions, it is possible that a displacement of this nature is not associated with increases in cation exchange capacity. With reference to the displacement, it is interesting to note from both tables 6 and 7 that, in general, the quantities of silica in the supernatant liquid from the deferrated residues are larger than those from the original colloids at

equivalent pH values and phosphate-ion concentrations. This mechanism of phosphate ion retention by soil colloids is being studied further.

The neutralization curves of the original and deferrated Colts Neck and the Penn colloids are presented graphically in figure 1. The usual procedure of plotting pH against cubic centimeters of alkali was not followed, since the purpose of this study was not to compare the neutralization curves of different colloids but to compare the curves of the original and deferrated residues of the same colloid. The curves were obtained, therefore, by plotting the alkali

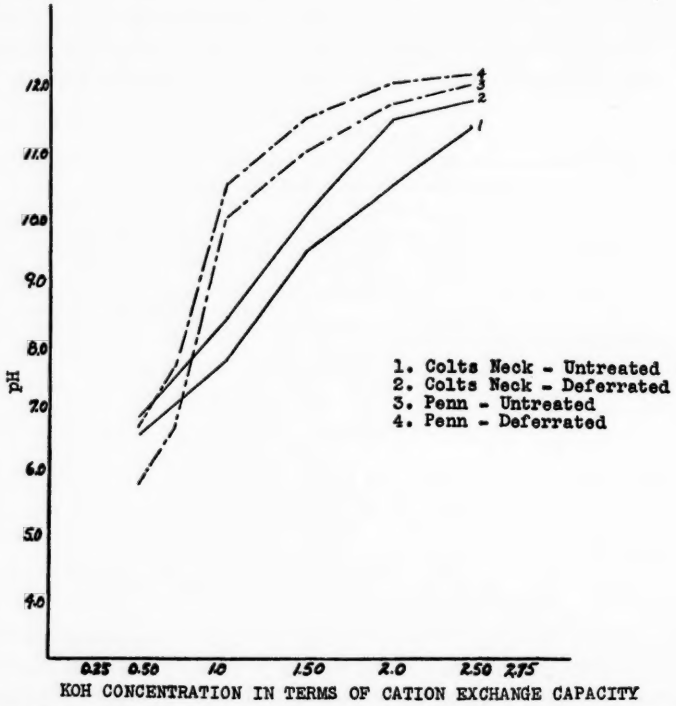


FIG. 1. NEUTRALIZATION CURVES OF UNTREATED AND DEFERRATED COLTS NECK AND PENN COLLOIDS

expressed in terms of the cation exchange capacity at pH 7.0 against the pH of the suspension, at equilibrium, of the H-colloids and the alkali. From figure 1, the outstanding feature of the curve for either colloid is that the neutralization curve of the deferrated colloid, though similar to the curve for the original colloid, is obtained at higher pH values. This behavior points to a definite reduction in the neutralization capacity of the deferrated residues for alkali. As previously pointed out, both the free iron oxides and the aluminosilicates are removed from the colloids by the iron oxide removal treat-

ment, and since both of these constituents can combine with alkali, the reduction in neutralization capacity can account for the variations between the curves. The curves for the Cecil and Sassafras colloids are essentially similar to those obtained for the Colts Neck and Penn, and are not, therefore, presented.

### EFFECT OF FREE IRON REMOVAL ON RETENTION AND RELEASE OF Ca, Ba, AND Mg BY ELECTRODIALYSIS

The changes in the properties of soil colloids produced as the result of the partial removal of the basoid fraction have been emphasized. It is the purpose of the following experiment to note whether or not alterations have resulted in the nature of the acidoid fraction of the colloids. Since the nature of the acidoid fraction influences to some extent the retention of specific cations by

TABLE 8

*Cation content and pH values of H-, Ca-, Mg-, and Ba-colloids*

| COLLOID | CATION CONTENT | | | pH VALUES (1:10 $H_2O$) | | | |
|---|---|---|---|---|---|---|---|
| | Ca | Mg | Ba | H | Ca | Mg | Ba |
| | *m.e./100 gm.* | *m.e./100 gm.* | *m.e./100 gm.* | | | | |
| *Untreated Colloids* | | | | | | | |
| Cecil | 11.05 | 10.40 | 9.40 | 5.30 | 7.20 | 6.00 | 6.40 |
| Colts Neck | 30.79 | 31.90 | 28.80 | 4.50 | 6.50 | 5.40 | 6.60 |
| Penn | 23.21 | 23.00 | 22.50 | 4.60 | 6.30 | 5.70 | 6.50 |
| Sassafras | 20.21 | 16.30 | 16.50 | 5.00 | 6.80 | 5.70 | 6.40 |
| *Deferrated Colloids* | | | | | | | |
| Cecil | 11.12 | 10.00 | 10.08 | 4.30 | 6.80 | 4.90 | 6.25 |
| Colts Neck | 31.29 | 32.90 | 28.80 | 4.30 | 6.90 | 4.50 | 6.50 |
| Penn | 23.21 | 21.50 | 19.80 | 4.45 | 6.90 | 5.30 | 6.35 |
| Sassafras | 21.76 | 16.50 | 22.50 | 4.35 | 6.20 | 4.80 | 6.30 |

colloids, it was believed that a study of the release of Ca, Mg, and Ba from cation-saturated colloids during electrodialysis would yield data concerned with changes in the acidoid fractions. With this object in view, the data in tables 8 and 9 are presented. The data in these tables differ in one main respect from the data in previous tables. For the purpose of comparing the release of cations during electrodialysis, equal quantities, with respect to the content of each specific cation, of the original and deferrated colloids were used rather than the weights of the residues of varying exchange capacity. The corrections for this variation were generally slight, with the exception of the Colts Neck colloid. In table 8, therefore, it should be noted that the exchangeable cation contents of the original and the deferrated colloids are approximately the same. Variations do exist in the degree of saturation of the colloids with specifications but it is believed that this is due, in part, to the technic

employed in preparing the cation-saturated colloids. The pH values must also be examined and compared on basis of the cation content and not on the basis of equal saturation.

It has been pointed out (6) that, in general, for cations fixed by colloids, the stability of the retained cation depends upon the silica-sesquioxide ratio. Less Mg is released during electrodialysis from colloids of high silica-sesquioxide ratio than from colloids of a low ratio (6). This is believed to be due to the greater stabilization of exchangeable Mg by colloids of a high silica content. Generally the pH values of Mg-saturated colloids are lower than the pH values of Ca-saturated colloids (1, 6) and this behavior is also advanced as a further proof of the stabilization of exchangeable Mg by soil colloids. The pH values of the colloids presented in table 8 afford excellent data for an examination of the foregoing theory.

Since the original and deferrated Sassafras colloids are only partially saturated with Mg and Ba, the data on these clays will be omitted from the following discussion.

The pH values of the cation-saturated colloids as presented in table 8 are of particular interest. The pH values of the Mg-saturated colloids are lower than those of the Ca- or Ba-saturated colloids by 0.4 to 1.2 pH unit with the original and from 1 to 2.4 pH units with the deferrated residues. This behavior is in line with the previously cited facts (1, 6). Only slight differences exist between the pH values of the Mg-saturated colloids in relation to the silica-sesquioxide ratio. This may, of course, be due to the fact that other acidoids (6) interfere in the evaluating of the correlation. Only slight differences are to be noted from the data in table 8 with respect to the pH values of the original Ca- and Ba-saturated colloids with the exception, however, of the pH of the original Cecil colloid. This colloid has a pH value about 0.8 higher for the Ca- than the Ba-saturated sample. The difference in pH between the deferrated residues and the original colloids is not very significant for the Ba-saturated samples. For the Ca-saturated samples, however, the difference is in favor of the residues with the Penn and Colts Neck and in favor of the original colloids with the Cecil and Sassafras. No relation exists between the silica-sesquioxide of the colloids and the pH values of the Ca- or Ba-colloids.

The release of cations by electrodialysis from the various colloids is presented in table 9, as well as the residual exchangeable cation content of the electrodialyzed colloids. The total recovery of cations by electrodialysis and salt displacement is also given in table 9. From the data concerned with the release of exchangeable Ca and Ba, it will be noted that only slight differences exist between the original and the deferrated colloids. The differences range from 0.6 per cent with the Cecil to 8.4 with the Penn colloid. With the Mg-colloids only slight differences exist between the original Penn and Sassafras and the deferrated residues of these two colloids, but significant variations do exist with the Colts Neck and Cecil colloids. The fact that less Mg is released from these two deferrated colloids by electrodialysis indicates a definite change

TABLE 9

*Release of cations by electrodialysis from untreated and deferrated colloids*

Time of electrodialysis, 24 hours for Ca and Ba; 48 hours for Mg

| COLLOID | CATIONS RELEASED BY ELECTRODIALYSIS | | | PER CENT OF TOTAL CATIONS RELEASED BY ELECTRODIALYSIS | | | CATIONS DISPLACED BY *N* $NH_4Ac$ AFTER ELECTRODIALYSIS | | | PER CENT OF TOTAL CATIONS DISPLACED BY *N* $NH_4Ac$ AFTER ELECTRODIALYSIS | | | RECOVERY OF CATIONS | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Ca | Ba | Mg | Ca | Ba | Mg | Ca | Ba | Mg | Ca | Ba | Mg | Ca | Ba | Mg |
| | *m.e./100 gm.* | *m.e./100 gm.* | *m.e./100 gm.* | | | | *m.e./100 gm.* | *m.e./100 gm.* | *m.e./100 gm.* | | | | *per cent* | *per cent* | *per cent* |
| *Untreated Colloids* | | | | | | | | | | | | | | | |
| Cecil | 10.06 | 8.60 | 8.00 | 91.0 | 95.5 | 76.9 | 0.99 | 0.59 | 0.92 | 9.0 | 2.2 | 8.8 | 100 | 97.7 | 85.7 |
| Colts Neck | 27.70 | 27.46 | 18.00 | 89.9 | 95.3 | 56.4 | 2.99 | 0.59 | 1.70 | 10.1 | 2.0 | 5.3 | 100 | 97.7 | 61.7 |
| Penn | 22.22 | 20.37 | 21.60 | 95.7 | 90.5 | 93.9 | 0.99 | 1.00 | 1.40 | 4.3 | 4.4 | 6.1 | 100 | 94.9 | 100 |
| Sassafras | 19.22 | 15.60 | 13.80 | 95.1 | 94.5 | 84.6 | 0.99 | 0.69 | 2.50 | 4.9 | 4.3 | 15.4 | 100 | 98.7 | 100 |
| *Deferrated Colloids* | | | | | | | | | | | | | | | |
| Cecil | 10.06 | 9.58 | 6.22 | 90.4 | 95.8 | 62.2 | 1.06 | 0.45 | 1.64 | 9.6 | 3.8 | 16.4 | 100 | 99.3 | 78.6 |
| Colts Neck | 28.50 | 25.94 | 14.96 | 91.1 | 90.0 | 45.4 | 2.79 | 0.45 | 1.22 | 8.9 | 1.6 | 3.8 | 100 | 91.6 | 49.2 |
| Penn | 22.31 | 19.20 | 19.06 | 96.1 | 98.9 | 88.6 | 0.89 | 0.40 | 1.40 | 3.9 | 1.1 | 6.5 | 100 | 100 | 95.1 |
| Sassafras | 20.86 | 21.60 | 14.74 | 95.8 | 96.0 | 89.3 | 0.90 | 0.59 | 1.00 | 4.2 | 3.6 | 6.0 | 100 | 99.6 | 95.3 |

in the nature of acidoid linkages. The removal of phosphorus and organic matter during the treatment of the colloids probably accounts for this variation, since the magnesium silicates are less soluble than compounds of magnesium with phosphoric or humic acids. The rate of release of Mg during electrodialysis is also less than that of either Ca or Mg, as the data in table 9 indicate. Complete recovery of Ca and Ba within experimental error from the colloids by electrodialysis and salt displacement indicates no fixation of these cations by the colloids. For Mg, however, definite indications of fixation were obtained with the Cecil and Colts Neck colloids, whereas a complete recovery was noted with the Penn and Sassafras colloids. Apparently, other factors besides the silica-sesquioxide ratio influence the release and fixation of Mg.

## SUMMARY

A study was made of the effects of free iron oxide removal from soil colloids upon certain properties of the colloids.

It was observed that the deferrated colloids lost more silica and sesquioxides during electrodialysis than did the untreated colloids. The composition of the sesquioxides varied, but the amount of aluminum electrodialyzed from the deferrated residues was always greater.

The removal of the free oxides lowered the ultimate pH of the colloids without materially affecting the cation exchange capacity at pH 7.0.

The removal of the free oxides decreased the adsorption of chloride and phosphate ions. Indications that soil colloids may adsorb phosphate ions without altering the cation exchange capacity were obtained with completely deferrated Colts Neck and Sassafras colloids.

The neutralization curves of the deferrated colloids were similar to the curves for the original colloids, but the residues possessed a lower buffer capacity.

The effect of free oxide removal upon the nature of the acidoid fraction was also studied. Only slight changes were observed, and these were ascribed to the removal of certain acidoids. In general, the release by electrodialysis of Ca and Ba from original and deferrated colloids was the same. Magnesium, however, differed from the former two cations in that less was released per unit time from the colloids by electrodialysis.

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# RELIABILITY OF THE PROPOSED SUCTION METHOD OF DETERMINING THE MOISTURE EQUIVALENT OF SOILS[1]

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The moisture equivalent is now rather generally recognized in this country as a fairly reliable measure of the texture of the soil (6) and "the moisture equivalent centrifuge is now a recognized piece of apparatus in well equipped soil physics laboratories" (5). In this laboratory the centrifuge has been in constant use for 25 years. As the apparatus is expensive and requires an electric current for its operation, there is a place for some method that will give the same values while requiring only simple equipment. To meet this need Bouyoucos (1, 2) has proposed a suction method requiring only such apparatus as is to be found in any chemical laboratory. He considers it rapid, accurate, and reliable, in addition to being simple and convenient. In his first paper describing the method (1) he gave no comparison of values obtained by the two methods, but in his second he reports such a comparison for 25 soils, the determinations by his method being made in his laboratory and the centrifuge being used with 13 of the soils at both Cornell University and the U. S. Department of Agriculture at Washington, 10 at only the former, and 2 at only the latter. Apparently, no determinations with the centrifuge were made in his own laboratory on the soils dealt with in his papers.

Shortly before the appearance of Bouyoucos' second paper a comparison of the two methods, using over one hundred soils, had been completed at this laboratory. The modifications of the suction method proposed in the second paper made a study of these desirable before the comparison was published, a study that circumstances have prevented until the present.

## MOISTURE EQUIVALENT DEFINED

The term *moisture equivalent* was introduced by Briggs and McLane in 1907 (3, p. 5) to designate "the percentage of water retained by a soil when the moisture content is reduced by means of a constant centrifugal force until it is brought to a state of capillary equilibrium with the applied force." At that time, using a centrifugal force 3,000 times gravity, they reported the moisture equivalents of 100 soils of known mechanical composition. Three years later (4) they adopted "as a standard basis of comparison a centrifugal force equal

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to 1,000 times the force of gravity, one centimeter as the thickness of the soil layer when packed in the cup and 40 minutes as the time of operation of the machine at the designated speed, 2440 r.p.m." They adopted 20°C. as the standard temperature but found the temperature effects so small that they can usually be disregarded.

Russel and Burr (5), as the result of a detailed study of "the effect of speed, period of centrifuging and depth of soil layer in the cup on the moisture equivalent . . . in an attempt to arrive at either a less empirical definition of the moisture equivalent or a better appreciation of the necessity of strict adherence to the present adopted procedure," concluded that "the moisture equivalent is best defined as Briggs and McLane have defined it, that is, centrifugal force 1000 g; depth of soil, 10 mm.; period, 40 minutes," remarking that "it is indeed fortunate that so near the desirable requirements were decided upon."

## DESCRIPTION OF THE SUCTION METHOD

The method described in Bouyoucos' first paper consists of connecting a small Buechner funnel with a suction flask, placing on this a filter paper, filling the funnel three-fourths full of air-dry soil, thoroughly soaking this with water, connecting the flask to the suction pipe on a faucet and then allowing the suction force from the running water to operate on the soil for about 12 minutes, the funnel being covered with a moist thick cloth, in order to prevent evaporation during the suction process. The funnel is then removed from the suction flask, the soil scraped into a weighing receptacle and its moisture content determined in the usual way. In his second paper he specifies a funnel 50 mm. in diameter and 25 mm. high, first passing the air-dry soil through a 2-mm. sieve, filling the funnel full and then compacting the soil by gently tapping the lower end of the funnel against the table. Also, instead of wetting the soil in the funnel by adding water to the surface, the filled funnel is placed in a beaker into which water is poured until it almost reaches the level of the top of the funnel and left for about 24 hours. The attempt is made to maintain in the funnel the same depth of wet soil, and not the same weight of dry soil, by levelling off the soil even with the top of the funnel after the soil has been suctioned for a minute and has become settled and compacted. The weight of the dry soil used in his determinations varied from about 40 to 85 gm., depending upon the class of soil. Suction is continued for 15 minutes after all the excess water on the top of the soil has disappeared, and during suction the soil is covered with a tumbler containing a moist cloth. The values obtained with a water jet pump giving a vacuum pressure of only about 15 mm. were similar to those found with a high vacuum pump, giving a vacuum of 4 mm.

## EXPERIMENTAL

In our comparison, 113 Minnesota soils selected from samples on hand at our laboratory so as to cover a wide range in moisture equivalent, were used.

They represent 15 different soil series in addition to muck and leafmold. About a third are surface samples, the others from various depths down to the sixth foot. The nitrogen content ranges from 0.001 to 2.03 per cent, and the pH value, from 4.7 to 8.1. The data on 23 representative soils are reported in table 3. The numbers in the first column indicate the order in moisture equivalent value, No. 1 being the soil with the lowest, 1.5, and No. 113 that with the highest, 90.6.

TABLE 1

*Effect of depth of soil in Buechner funnel upon the amount of water retained after suction*

| REFERENCE NUMBER | WATER RETAINED* AFTER SUCTION | | | DIFFERENCE BETWEEN DUPLICATE DETERMINATIONS WITH SOIL DEPTH OF | |
|---|---|---|---|---|---|
| | With soil depth of | | Excess with deeper layer | | |
| | 20 mm. | 25 mm. | | 20 mm. | 25 mm. |
| | *per cent* | *per cent* | *per cent* | *per cent* | *per cent* |
| 1 | 4.2 | 4.5 | 0.3 | 0.7 | 0.9 |
| 2 | 4.3 | 4.7 | 0.4 | 0.7 | 0.6 |
| 3 | 3.6 | 3.9 | 0.3 | 0.1 | 0.1 |
| 4 | 4.0 | 5.1 | 1.1 | 0.2 | 0.2 |
| 5 | 4.4 | 6.2 | 1.8 | 0.0 | 0.9 |
| 6 | 4.2 | 4.6 | 0.4 | 0.3 | 0.4 |
| 7 | 4.8 | 9.2 | 4.4 | 0.6 | 0.5 |
| 8 | 4.6 | 7.6 | 3.0 | 0.2 | 1.1 |
| 9 | 6.7 | 7.1 | 0.4 | 1.7 | 2.2 |
| 35 | 18.6 | 16.8 | −1.8 | 1.2 | 1.1 |
| 36 | 18.9 | 24.1 | 5.2 | 0.8 | 0.4 |
| 40 | 20.3 | 19.5 | −0.8 | 0.3 | 1.9 |
| 44 | 24.8 | 27.8 | 3.0 | 0.0 | 0.9 |
| 61 | 32.5 | 31.0 | −1.5 | 0.8 | 1.4 |
| 65 | 33.1 | 30.1 | −3.0 | 1.7 | 0.6 |
| 71 | 36.3 | 39.9 | 3.6 | 1.9 | 1.3 |
| 80 | 36.6 | 39.7 | 3.1 | 3.6 | 2.2 |
| 84 | 39.7 | 40.9 | 1.2 | 1.7 | 0.1 |
| 87 | 42.2 | 43.6 | 1.4 | 5.2 | 0.2 |
| 95 | 48.5 | 48.6 | 0.1 | 0.3 | 1.2 |
| Average.... | 19.6 | 20.7 | 1.1 | 1.1 | 0.9 |

* Average of duplicate determinations.

Though the Buechner funnel used was of the size specified in Bouyoucos' second paper, the depth of the air-dry soil placed in it was that specified in his first paper, approximately 20 mm. ("funnel three-fourths full"). The soil was moistened, as described in the first paper, by adding water to the surface at intervals of 10 to 15 minutes after it had begun to drain from the funnel and not by allowing the funnel to stand in a beaker of water. A few of the soils very rich in organic matter absorbed water so slowly that with them the addition was continued at intervals for an hour or more. The determinations

by the centrifuge as well as by suction were made in duplicate. With the latter where the values were widely discordant a third or even a fourth determination was made and the average of the closest two reported in the table.

In order to decide whether the proposed increase in depth of the air-dry soil in the funnel or the change in method of wetting it would lessen or increase the amount of water retained after suction, we have now repeated the determinations on 20 of the soils, using the 9 coarsest in texture and 11 of finer texture

TABLE 2

*Effect of method of wetting the soil in Buechner funnel upon amount of water retained after suction*

| REFERENCE NUMBER | WATER RETAINED* AFTER SUCTION | | | DIFFERENCE BETWEEN DUPLICATE DETERMINATIONS WHEN SOIL WAS WETTED FROM | |
|---|---|---|---|---|---|
| | Soil wetted from | | Excess with latter | | |
| | Above for 15 min. | Below for 7 hours | | Above for 15 min. | Below for 7 hours |
| | *per cent* | *per cent* | *per cent* | *per cent* | *per cent* |
| 1 | 4.5 | 4.1 | −0.4 | 0.9 | 0.0 |
| 2 | 4.7 | 5.8 | 1.1 | 0.6 | 1.0 |
| 3 | 3.9 | 4.3 | 0.4 | 0.1 | 3.1 |
| 4 | 5.1 | 3.8 | −1.3 | 0.2 | 0.3 |
| 5 | 6.2 | 6.4 | 0.2 | 0.9 | 0.8 |
| 6 | 4.6 | 6.6 | 2.0 | 0.4 | 2.4 |
| 7 | 9.2 | 8.3 | −0.9 | 0.5 | 0.4 |
| 8 | 7.6 | 8.1 | 0.5 | 1.1 | 0.9 |
| 9 | 7.1 | 9.0 | 1.9 | 2.2 | 4.3 |
| 35 | 16.8 | 17.6 | 0.8 | 1.1 | 2.2 |
| 36 | 24.1 | 24.8 | 0.7 | 0.4 | 2.0 |
| 40 | 19.5 | 18.6 | −0.9 | 1.9 | 0.4 |
| 44 | 27.8 | 25.8 | −2.0 | 0.9 | 3.1 |
| 61 | 31.0 | 35.8 | 4.8 | 1.4 | 5.2 |
| 65 | 30.1 | 32.3 | 2.2 | 0.6 | 4.9 |
| 71 | 39.9 | 44.1 | 4.2 | 1.3 | 6.1 |
| 80 | 39.7 | 45.2 | 5.5 | 2.2 | 3.0 |
| 84 | 40.9 | 41.7 | 0.8 | 0.1 | 3.1 |
| 87 | 43.6 | 44.8 | 1.2 | 0.2 | 1.7 |
| 95 | 48.6 | 47.8 | −0.8 | 1.2 | 1.0 |
| Average.... | 20.8 | 21.7 | 0.9 | 1.0 | 2.3 |

* Average of duplicate determinations.

which had shown the greatest difference between moisture equivalent and suction value. Neither the values obtained with the thicker layer of soil (table 1) nor those from the prolonged wetting (table 2) depart definitely from those obtained earlier. We conclude, therefore, that the suction values of the remaining 93 soils would not have been significantly different had they been determined with a greater depth in the funnel and kept in contact with water over night.

The term *suction value* is used in this paper merely to differentiate in the discussion the amount of water retained by a soil when subjected to the treatment described in Bouyoucos' second paper from the value obtained with the centrifuge and not with the idea of introducing it into soils literature. If the values obtained by the suction method are the same as those from the centrifugal method they also may properly be referred to as *moisture equivalents*,

TABLE 3

*"Suction values" of representative soils compared with their moisture equivalents*

| REFERENCE NUMBER | COUNTY | SOIL SERIES | DEPTH OF SECTION | N | pH | MOISTURE EQUIVALENT | SUCTION VALUE | EXCESS OF MOISTURE BY SUCTION | RATIO OF SUCTION VALUE TO MOISTURE EQUIVALENT | DIFFERENCE BETWEEN DUPLICATE DETERMINATIONS | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | Moisture equivalent | Suction value |
| | | | *inches* | *per cent* | | | *per cent* | *per cent* | | *per cent* | *per cent* |
| 1 | Aitkin | Grayling | 61–72 | 0.001 | 5.7 | 1.5 | 4.2 | 2.7 | 2.80 | 0.0 | 0.7 |
| 7 | Aitkin | Grayling | 13–24 | 0.010 | 5.5 | 2.9 | 4.8 | 1.9 | 1.66 | 0.1 | 0.6 |
| 8 | Aitkin | Rabey | 25–36 | 0.014 | 5.6 | 3.0 | 4.6 | 1.6 | 1.53 | 0.2 | 0.2 |
| 11 | Aitkin | Munger | 61–72 | 0.001 | 6.4 | 4.8 | 6.1 | 1.3 | 1.27 | 0.1 | 1.0 |
| 15 | Aitkin | Cloquet | 13–24 | 0.017 | 5.1 | 6.0 | 7.0 | 1.0 | 1.17 | 0.6 | 0.8 |
| 18 | Aitkin | Swan | 25–36 | 0.008 | 7.5 | 7.2 | 9.2 | 2.0 | 1.28 | 0.1 | 0.5 |
| 22 | Aitkin | Hill City | 1–6 | 0.061 | 5.0 | 9.0 | 9.8 | 0.8 | 1.09 | 0.3 | 1.9 |
| 31 | Aitkin | Kingsley | 3–19 | 0.049 | 5.0 | 12.8 | 14.6 | 1.8 | 1.14 | 0.1 | 1.4 |
| 37 | Lac qui Parle | Moody | 1–6 | 0.198 | 6.2 | 16.0 | 21.0 | 5.0 | 1.31 | 0.4 | 1.9 |
| 45 | Jackson | Bremer | 13–24 | 0.090 | 6.7 | 21.2 | 22.5 | 1.3 | 1.06 | 0.6 | 5.0 |
| 50 | Houston | Tama | 13–24 | 0.056 | 5.2 | 23.1 | 28.5 | 5.4 | 1.23 | 0.2 | 0.3 |
| 52 | Rice | Carrington | 1–6 | 0.263 | 5.5 | 24.0 | 27.0 | 3.0 | 1.12 | 1.2 | 0.1 |
| 54 | Fillmore | Tama | 13–24 | 0.049 | 5.4 | 25.1 | 28.4 | 3.3 | 1.13 | 1.0 | 5.9 |
| 57 | Nobles | Clarion | 1–12 | 0.270 | 5.8 | 25.9 | 28.5 | 2.6 | 1.10 | 0.7 | 5.9 |
| 63 | Rice | Carrington | 7–12 | 0.278 | 5.3 | 28.2 | 32.5 | 4.3 | 1.15 | 0.1 | 1.3 |
| 78 | Lac qui Parle | Barnes | 25–36 | 0.093 | 8.0 | 31.9 | 35.6 | 3.7 | 1.12 | 1.4 | 0.4 |
| 87 | Lac qui Parle | Barnes | 7–12 | 0.264 | 6.1 | 37.0 | 42.2 | 5.2 | 1.14 | 0.3 | 5.2 |
| 98 | Lac qui Parle | Webster | 1–6 | 0.521 | 6.5 | 43.8 | 45.5 | 1.7 | 1.04 | 0.0 | 0.3 |
| 101 | Aitkin | Leafmold | 1 | 0.588 | 5.3 | 46.9 | 54.1 | 7.2 | 1.15 | 0.1 | 7.3 |
| 103 | Jackson | Muck | 7–12 | 1.250 | 3.9 | 48.3 | 58.1 | 9.8 | 1.20 | 0.3 | 13.8 |
| 104 | Jackson | Muck | 24–36 | 0.861 | 5.6 | 49.9 | 49.6 | −0.3 | 0.99 | 0.2 | 1.8 |
| 108 | Aitkin | Leafmold | 1 | 0.966 | 5.8 | 52.6 | 62.3 | 9.7 | 1.19 | 1.1 | 4.4 |
| 113 | Aitkin | Leafmold | 1 | 2.030 | 4.1 | 90.6 | 98.3 | 7.7 | 1.08 | 0.3 | 6.0 |

but, if not, some other designation should be found for them, because the definition of the term given by Briggs and McLane has become generally recognized.

With the coarsest sands, having moisture equivalents below 2.0, the suction value is more than twice as high as the moisture equivalent, and with soils of somewhat finer texture it is from 30 to 60 per cent higher. With the soils having moisture equivalents higher than 8.0 the ratio shows no distinct de-

TABLE 4

*Comparison of authors' data with those reported by Bouyoucos*

| GROUP | RANGE IN MOISTURE EQUIVALENT IN GROUPS | NUMBER OF SAMPLES REPORTED | | AVERAGE RATIO: SUCTION VALUE TO MOISTURE EQUIVALENT | | RANGE IN RATIO: SUCTION VALUE TO MOISTURE EQUIVALENT | |
|---|---|---|---|---|---|---|---|
| | | Pinckney | Bouyoucos | Pinckney | Bouyoucos | Pinckney | Bouyoucos |
| 1 | 1.0– 2.0 | 6 | 0 | 2.46 | .... | 2.80–2.12 | ..... |
| 2 | 2.1– 6.0 | 10 | 1 | 1.26 | 2.21 | 1.67–1.17 | ..... |
| 3 | 6.1– 8.0 | 4 | 0 | 1.33 | .... | 1.49–1.23 | ..... |
| 4 | 8.1–12.0 | 10 | 1 | 1.17 | 1.34 | 1.36–1.09 | ..... |
| 5 | 12.1–16.0 | 9 | 3 | 1.27 | 0.99 | 1.31–1.04 | 1.07–0.91 |
| 6 | 16.1–20.0 | 5 | 1 | 1.18 | 0.91 | 1.26–1.10 | ..... |
| 7 | 20.1–25.0 | 9 | 2 | 1.10 | 1.04 | 1.23–1.01 | 1.09–0.99 |
| 8 | 25.1–30.0 | 12 | 7 | 1.09 | 1.07 | 1.20–0.95 | 1.14–1.02 |
| 9 | 30.1–35.0 | 20 | 2 | 1.09 | 1.15 | 1.16–0.99 | 1.18–1.12 |
| 10 | 35.1–40.0 | 7 | 2 | 1.11 | 1.10 | 1.18–1.05 | 1.11–1.10 |
| 11 | 40.1–45.0 | 6 | 1 | 1.06 | 1.03 | 1.14–0.98 | ..... |
| 12 | 45.1–50.0 | 6 | 1 | 1.10 | 1.14 | 1.20–0.99 | ..... |
| 13 | 50.1–55.0 | 4 | 1 | 1.19 | 1.11 | 1.24–1.17 | ..... |
| 14 | 55.0–91.0 | 5 | 3 | 1.09 | 1.08 | 1.15–1.03 | 1.10–1.06 |

TABLE 5

*Relation of suction value-moisture equivalent ratio to nitrogen content of the soil*

| GROUP I | | | | GROUP II | | | | GROUP III | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Reference number | N | Ratio | Moisture equivalent | Reference number | N | Ratio | Moisture equivalent | Reference number | N | Ratio | Moisture equivalent |
| | *per cent* | | | | *per cent* | | | | *per cent* | | |
| 61 | .031 | 1.20 | 27.0 | 45 | .090 | 1.06 | 21.2 | 53 | .270 | 1.09 | 25.0 |
| 75 | .031 | 1.03 | 31.4 | 78 | .093 | 1.12 | 31.9 | 57 | .270 | 1.10 | 25.9 |
| 77 | .032 | 1.09 | 31.4 | 69 | .126 | 1.07 | 30.7 | 59 | .275 | 0.95 | 26.3 |
| 74 | .036 | 1.12 | 31.3 | 71 | .136 | 1.15 | 30.8 | 63 | .278 | 1.15 | 28.2 |
| 70 | .037 | 1.08 | 30.8 | 64 | .142 | 1.05 | 29.0 | 49 | .288 | 1.01 | 22.7 |
| 79 | .038 | 1.14 | 31.9 | 72 | .148 | 1.11 | 30.8 | 84 | .297 | 1.16 | 34.1 |
| 65 | .044 | 1.14 | 29.0 | 76 | .151 | 1.16 | 31.4 | 55 | .301 | 1.05 | 25.8 |
| 62 | .045 | 1.19 | 27.8 | 90 | .160 | 1.10 | 38.0 | 81 | .314 | 1.04 | 33.1 |
| 50 | .056 | 1.23 | 23.1 | 47 | .174 | 1.11 | 21.9 | 67 | .318 | 1.11 | 30.1 |
| 82 | .057 | 1.00 | 33.1 | 80 | .211 | 1.12 | 32.6 | 68 | .318 | 1.12 | 30.6 |
| 92 | .060 | 1.09 | 39.3 | 73 | .220 | 1.05 | 31.1 | 88 | .355 | 1.05 | 37.0 |
| 58 | .066 | 1.09 | 26.0 | 46 | .261 | 1.04 | 21.8 | 83 | .400 | 0.99 | 34.1 |
| 51 | .072 | 1.19 | 24.0 | 85 | .262 | 1.07 | 35.0 | 86 | .466 | 1.18 | 36.1 |
| 60 | .076 | 1.14 | 27.0 | 52 | .263 | 1.12 | 24.0 | 54 | .490 | 1.13 | 25.1 |
| 48 | .084 | 1.03 | 22.0 | 87 | .264 | 1.14 | 37.0 | 91 | .606 | 1.06 | 38.8 |
| | | | | | | | | 89 | .669 | 1.13 | 38.0 |

*Summary*

| GROUP | NUMBER OF SAMPLES | RANGE IN PERCENTAGE OF N | RATIO | |
|---|---|---|---|---|
| | | | Average | Range |
| I | 15 | .031–.084 | 1.12 | 1.03–1.23 |
| II | 15 | .090–.264 | 1.10 | 1.04–1.16 |
| III | 16 | .270–.669 | 1.08 | 0.95–1.18 |

pendence upon the texture, averaging about 1.1 for the 93 soils; 19 of these, approximately a fifth, show a ratio within the limits 1.05 to 0.95, or a departure of suction value from moisture equivalent of not more than 5 per cent. In table 4 our data are summarized in comparison with those reported by Bouyoucos (2, p. 168).

TABLE 6

*Relation of suction value-moisture equivalent ratio to reaction of the soil*

| GROUP I | | | | GROUP II | | | | GROUP III | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Reference number | pH value | Ratio | Moisture equivalent | Reference number | pH value | Ratio | Moisture equivalent | Reference number | pH value | Ratio | Moisture equivalent |
| 50 | 5.2 | 1.23 | 23.1 | 45 | 6.7 | 1.06 | 21.2 | 62 | 7.5 | 1.19 | 27.8 |
| 51 | 5.2 | 1.19 | 24.0 | 53 | 6.7 | 1.09 | 25.0 | 48 | 7.6 | 1.03 | 22.0 |
| 63 | 5.3 | 1.15 | 28.2 | 86 | 6.7 | 1.18 | 36.1 | 73 | 7.6 | 1.05 | 31.1 |
| 54 | 5.4 | 1.13 | 25.1 | 72 | 7.0 | 1.11 | 30.8 | 76 | 7.6 | 1.16 | 31.4 |
| 52 | 5.5 | 1.12 | 24.0 | 82 | 7.0 | 1.00 | 33.1 | 88 | 7.6 | 1.05 | 37.0 |
| 89 | 5.5 | 1.13 | 38.0 | 59 | 7.1 | 0.95 | 26.3 | 60 | 7.8 | 1.14 | 27.0 |
| 57 | 5.8 | 1.10 | 25.9 | 67 | 7.1 | 1.11 | 30.1 | 71 | 7.8 | 1.15 | 30.8 |
| 56 | 5.9 | 0.99 | 25.9 | 68 | 7.1 | 1.12 | 30.6 | 80 | 7.8 | 1.12 | 32.6 |
| 46 | 6.0 | 1.04 | 21.8 | 58 | 7.2 | 1.09 | 26.0 | 77 | 7.9 | 1.09 | 31.4 |
| 55 | 6.0 | 1.05 | 25.8 | 79 | 7.2 | 1.14 | 31.9 | 65 | 8.0 | 1.14 | 29.0 |
| 47 | 6.1 | 1.11 | 21.9 | 91 | 7.2 | 1.06 | 38.8 | 74 | 8.0 | 1.12 | 31.3 |
| 87 | 6.1 | 1.14 | 37.0 | 83 | 7.3 | 0.99 | 34.1 | 78 | 8.0 | 1.12 | 31.9 |
| 81 | 6.2 | 1.04 | 33.1 | 90 | 7.3 | 1.10 | 38.0 | 92 | 8.0 | 1.09 | 39.3 |
| 84 | 6.4 | 1.16 | 34.1 | 64 | 7.4 | 1.05 | 29.0 | 70 | 8.1 | 1.08 | 30.8 |
| 49 | 6.6 | 1.01 | 22.7 | 85 | 7.4 | 1.07 | 35.0 | 75 | 8.1 | 1.03 | 31.4 |
| 69 | 6.6 | 1.07 | 30.7 | 61 | 7.5 | 1.20 | 27.0 | .. | ... | .... | .... |

*Summary*

| GROUP | NUMBER OF SAMPLES | pH RANGE | RATIO | |
|---|---|---|---|---|
| | | | Average | Range |
| I | 16 | 5.2–6.6 | 1.10 | 0.99–1.23 |
| II | 16 | 6.7–7.5 | 1.08 | 0.95–1.20 |
| III | 15 | 7.5–8.1 | 1.10 | 1.03–1.19 |

Duplicate determinations are far less concordant with the suction method than with the centrifuge, the average difference between duplicates for the 113 soils being 0.5 for the centrifuge and 2.3 for the suction method.

In order to bring out any relation there may be of the $\frac{\text{suction value}}{\text{moisture equivalent}}$ to either the organic matter content or the reaction, all the soils having moisture equivalents between 20 and 40 have been arranged in table 5 in order of increasing nitrogen content and in table 6 in order of rising pH value. Nitrogen was determined by the Kjeldahl method, and the reaction, by means of a pH meter using a glass electrode. No dependence of the ratio upon the nitro-

gen content is to be seen. For the present purposes the organic matter may be assumed to vary as the nitrogen, being about 20 times as high as the latter in the surface soils and somewhat lower in the subsurface samples. Of the 46 soils 16 are from the surface 1 to 6 inches, 9 from the second 6 inches, 9 from the second foot, and the 13 others from the third, fourth, fifth, or sixth foot. A similar lack of dependence of the ratio upon the reaction is seen in table 6. The average ratio is 1.10 for the most acid group of 16, with pH values between 5.2 and 6.6, as well as for the alkaline group of 15, with pH values from 7.5 to 8.1.

## DISCUSSION

Bouyoucos' conclusion (2) that his suction method is simple, convenient, easily available, requires no expensive equipment, and will be useful where no moisture equivalent centrifuge is available is fully justified, as is also his statement that "in some soils the two methods agree very closely, whereas in others the agreement is not so close. . . . In general the suction method tends to give somewhat higher values than the centrifuge method." We find duplicate determinations by the suction method to agree much less closely than do those with the centrifuge. This may be illustrated by the data on the 49 soils with moisture equivalents between 20 and 40:

| DIFFERENCE BETWEEN DUPLICATES, IN PERCENTAGE OF WATER RETAINED | NUMBER OF SOILS WITH THIS DIFFERENCE | |
|---|---|---|
| | With centrifuge | By suction |
| 0.0–0.5 | 35 | 5 |
| 0.6–1.0 | 12 | 9 |
| 1.1–2.0 | 2 | 11 |
| 2.1–4.0 | 0 | 12 |
| 4.1–6.9 | 0 | 12 |

For comparing the relative texture of soils within one area, as for instance the samples from different horizons of 10 to 50 soil types within one county, the suction method may prove about as satisfactory as the centrifuge; but for comparing these data with those from another area, on the samples from which the centrifuge had been used, it is less satisfactory; and for sands, unless further developed, is quite unsuitable.

In a laboratory where a moisture equivalent centrifuge and an experienced operator are available the suction method will cause delay instead of saving time and labor, except possibly where a determination on only a very few samples at one time is desired. Where persons interested in soil texture do not have convenient access to the service of a centrifuge the suction method is to be commended.

## SUMMARY

In a comparison of the value obtained by Bouyoucos' proposed suction method with the moisture equivalent determined with the Briggs-McLane

centrifuge, 113 Minnesota soils were used. These ranged in moisture equivalent from 1.5 to 90.6, in nitrogen from 0.001 to 2.03 per cent, and in pH from 4.7 to 8.1. Though the relationships found are in general agreement with those reported by Bouyoucos, they do not fully support his conclusions as to the reliability and general desirability of the method.

With loams and soils of still finer texture the suction value averaged about one-tenth higher than the moisture equivalent, whereas with the individual soils it varied from practically equal to one-third higher. With the sands of coarsest texture it was twice as high, or still higher, and with soils intermediate between these and loams it was generally intermediate but widely variable. Duplicate determinations by the suction method were found much less concordant than those with the centrifuge.

Only where a moisture equivalent centrifuge is not available does the use of the suction method appear desirable, and the values so obtained should be referred to by some other designation than *moisture equivalent.*

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# WILTING COEFFICIENT AND WILTING PERCENTAGE OF THREE FOREST SOILS OF THE DUKE FOREST

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Soil analyses and determinations of moisture quivalent, wilting coefficient, and wilting percentage were made in connection with studies of the edaphic factors influencing root development in three soils of the Duke Forest, Durham, North Carolina. The soils are Congaree clay loam, Georgeville clay, and Orange loam. Certain moisture relations in the three soils are clarified by the data obtained.

The Congaree clay loam area that was studied occupies the forested bottom land along Browning's Branch in Compartment 32 and 33 of the Durham Division of the Duke Forest. The area is subject to inundation but between overflows is moderately well drained. Variable amounts of alluvium are deposited with each inundation. The soil has developed only an $A_1$ horizon. It consists of a brown clay loam that is plastic when moist, extending to a depth of 1.5 to 2.5 inches. From that depth down to 6.5 to 10 inches is a layer of structureless sandy loam, lighter in color, largely because of a lower organic matter content. Below the latter layer are generally found stratified layers of sand and sandy loam. At a depth of 18 to 20 inches may be found residual soil, or, 3 or more additional feet of alluvial soil and then residual soil. Most of the entire soil is rather loose and contains small mica flakes.

The Georgeville clay area is in Stand 2, Compartment 15 of the New Hope Creek Division of the Forest. The area is well drained, being located on a gently rounded upland area between two small streams, and is forested with an oak-hickory wood. The Georgeville soil profile is well developed. The $A_1$ horizon is light brown, stony clay, passing at 0.5 to 1.5 inches into the $A_2$ horizon, a reddish yellow, rather compact, stony clay exhibiting a crumb structure and extending to a depth of 5 to 7 inches. The $B_1$ horizon is dark red, brittle clay, exhibiting bright surfaces when moist soil is broken. It gradually grades into the $B_2$ horizon, which is slightly darker and more compact than the $B_1$. The C horizon occurs at a depth of 24 to 40 inches. It is yellowish red granular clay with lighter mottlings.

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The Orange loam area is in Stand 1 of Compartment 15 of the New Hope Creek Division of the Forest. The area is upland but is poorly drained, the slope being too gentle to allow proper drainage. The forest on the area is of the post oak-blackjack oak type. The $A_1$ horizon is light gray, floury, stony loam, underlain at 1 to 2 inches by dull yellow loam ($A_2$ horizon) which extends to a depth of 7 to 9 inches. The B horizon is dingy yellow, extremely plastic, waxy, impervious clay. The C horizon, which begins at 17 to 20 inches, is friable, mixed green and yellow clay.

## METHODS OF STUDY

Following preliminary examination with a soil auger, three locations were selected as typical of each area. At each location a profile was dug, and duplicate pint samples of soil were taken from each soil horizon by means of a soil sampler devised to take samples of undisturbed soil for making determinations of wilting percentage (4). At regularly spaced intervals near the center of each area, 20 samples in duplicate were taken from the surface horizon. The subsurface soil samples were taken as the soil profiles were dug. The soil from each profile was removed to the desired depth for sampling (2 inches, 10 inches, and 24 inches). At regular intervals across the exposed surface three samples in duplicate were taken from each profile.

A second set of samples was collected in loose condition from the profiles, 2 quarts being taken from each horizon. These samples were brought into the laboratory, dried, and crushed with a wooden rolling pin so as to break up the soil aggregates and enable them to pass through a 2-mm. sieve. Soil particles that did not pass through the sieve openings were classified as gravel. The portion of the soil that passed through the sieve was then thoroughly mixed and all but 1 quart discarded. In each soil a composite sample was then made for each horizon by combining samples from the profiles. The composite samples were used for mechanical analyses, which were made in duplicate by a modification of Olmstead's (7) pipette method. The textural class for each horizon was determined by the equilateral triangle method of Davis and Bennett (6).

The samples for determination of the wilting percentage were obtained in cardboard containers that had been boiled in paraffin to make them moisture-proof. One series of these duplicate pint samples was used in the determination of the wilting percentage. The procedure for the determination of the lower limit of available soil moisture as outlined and discussed by Briggs and Shantz (1, 3) was used as a basis for determination of the wilting percentage. All soil down to within 1.5 inches of the top of each container was removed, leaving a sample approximately 1.5 inches thick in each can. The soil in the containers was saturated with tap water, and germinating oat (*Avena sativa* L.) seeds were placed on the surface. These seeds were covered with approximately one-fourth inch of dry, finely divided soil taken from the portion of soil that had been removed from the top. This covering of soil was allowed to

become moistened by capillary rise of water from the saturated soil below. The samples were placed in the Duke University greenhouse. The surface of the soil was kept moist by additions of small amounts of water until the plants had become well established and the moisture content of the soil below the surface had been reduced to near the wilting percentage. At that time the surface of the soil was sealed with paraffin to a depth of about 5 mm. When the oats were permanently wilted the moisture content was determined for the entire soil sample except that part which had been used as a cover for the oats seeds.

Oats were used as phytometers because previous experiments conducted in the Duke University greenhouse showed that good growth of root and tops is obtained from them and their wilting point can be easily ascertained. The work of Veihmeyer and Hendrickson (8, 9, 10) indicates that any small plant grown in a sealed container may be used as an indicator of the minimum available moisture content.

The second series of pint samples was used to determine moisture equivalent. The samples were carefully removed from the containers, and blocks of soil one-half inch thick were cut to fit the moisture equivalent pans. One block was cut from the upper part of a sample and another 1 inch from the bottom of the first block. Data from these determinations are designated as "moisture equivalent in place" for each particular depth in the soil profile from which the one-half inch thick soil samples were taken. The remainder of each pint sample was rolled and run through a 2-mm. sieve. The gravel was discarded. The portion that passed through the sieve was then thoroughly mixed, and duplicate samples were taken for determining the moisture equivalent of the sieved soil. The usual procedure in determining the moisture equivalent as given by Veihmeyer, Israelson, and Conrad (12) was followed. The wilting coefficient values for the samples were determined from the moisture equivalent values in place. The relationship between the moisture equivalent and the wilting coefficient of soils as developed by Briggs and Shantz (2) was used to determine the wilting coefficients.

## RESULTS

The results of the mechanical analyses of the Congaree, Georgeville, and Orange soils are presented in table 1. On the basis of the texture of the $A_1$ horizon it is apparent that the Congaree soil is classed as a clay loam, the Georgeville as a clay, and the Orange as a loam.

The profile of the Congaree soil is almost entirely free of gravel. In the A horizons of the Georgeville and the $A_2$ horizon of the Orange the gravel constitutes approximately one-fourth of the total weight of soil. The other horizons in the latter two soils are less gravelly. The Georgeville soil contains more gravel throughout the profile than the other two soils.

In regard to the other soil fractions, the Congaree is characterized by relatively large percentages of sand and small amounts of colloids at all depths

and by a decreasing clay content with increased depth. The high sand content of horizon 4 should be noted. Both the A horizons of the Georgeville soil are composed of approximately equal amounts of total sands, silt, and clay. The B horizons are very high in clay content and percentage of colloids. In the Orange soil the A horizons are composed largely of equal amounts of total sands and silt, whereas the B horizons are composed of about 50 per cent clay.

If all the means of the moisture equivalent determinations made at the various depths are considered, a rather complete picture is obtained of moisture equivalent values throughout most of each of the three soil profiles. The

TABLE 1

*Results, in percentage of oven-dry weight, of mechanical analyses of Congaree, Georgeville, and Orange soils determined by the pipette method*

| HORIZON | DEPTH, INCHES | GRAVEL | TOTAL SANDS | SILT | CLAY | COLLOIDS | TEXTURAL GRADE |
|---|---|---|---|---|---|---|---|
| | | | *Congaree soil* | | | | |
| $A_1$ | 0–2 | None | 38.42 | 37.81 | 23.77 | 15.68 | Clay loam |
| 2 | 2–7.5 | None | 57.75 | 24.45 | 17.00 | 14.74 | Sandy loam |
| 3 | 7.5–19 | Trace | 58.21 | 25.49 | 16.30 | 11.54 | Sandy loam |
| 4 | 19–25 | Trace | 84.80 | 8.81 | 6.39 | 4.26 | Sand |
| 5 | Below 25 | None | 57.36 | 27.78 | 14.86 | 10.32 | Sandy loam |
| | | | *Georgeville soil* | | | | |
| $A_1$ | 0–1.2 | 27.96 | 29.33 | 38.11 | 32.56 | 21.60 | Clay |
| $A_2$ | 1.2–6 | 22.78 | 27.85 | 37.22 | 34.93 | 26.00 | Clay |
| $B_1$ | 6–20 | 9.32 | 10.77 | 16.51 | 72.72 | 70.53 | Clay |
| $B_2$ | 20–33 | 4.85 | 9.15 | 22.36 | 68.49 | 64.96 | Clay |
| C | Below 33 | Trace | 17.05 | 31.69 | 51.26 | 41.98 | Clay |
| | | | *Orange soil* | | | | |
| $A_1$ | 0–1.5 | 4.78 | 40.64 | 43.98 | 15.38 | 11.02 | Loam |
| $A_2$ | 1.5–8 | 27.07 | 42.94 | 38.76 | 18.30 | 15.46 | Loam |
| $B_1$ | 8–14 | 3.18 | 18.71 | 30.39 | 50.90 | 46.38 | Clay |
| $B_2$ | 14–19 | 1.47 | 21.51 | 30.67 | 47.82 | 40.80 | Clay |
| C | Below 19 | 1.79 | 35.98 | 34.00 | 30.02 | 26.36 | Clay |

results are presented in table 2. It is evident that the moisture equivalent values are high near the surface of the soils.

In the Congaree profile the values drop off rapidly with increasing depth, reaching a minimum in horizon 3 at a depth of 12 to 16 inches. The values are slightly higher in horizons 4 and 5 but are still relatively low. The fact that the amounts of colloids determined by the pipette method (table 1) are increasingly smaller until the lowest horizon is reached, where the amount increases again, partly accounts for the moisture equivalent values that were obtained, because soils with high percentages of the smaller soil fractions retain more moisture when centrifuged than do those with low percentages.

In the Georgeville profile the moisture equivalent drops to a minimum in the $A_2$ horizon at a depth of approximately 2 to 4 inches but because of the extremely high amount of colloids in the B horizons, the moisture equivalent rises to a high level. The maximum moisture equivalent, 38 per cent, is at a depth of 23 inches.

In the Orange profile approximately the same situation exists as in the Georgeville. The moisture equivalents, however, are generally lower, as might be expected upon consideration of the amounts of colloids (table 1), which are correspondingly lower.

TABLE 2

*Mean moisture equivalents with standard errors determined from pint samples taken at the depths indicated in the Congaree, Georgeville, and Orange soil profiles*

| SAMPLE | CONGAREE | GEORGEVILLE | ORANGE |
|---|---|---|---|
| Surface* (0–3 inches) | | | |
| In place (top) | 33.56 ±0.98 | 26.70 ±0.54 | 21.46 ±0.77 |
| In place (1.5 inches) | 25.87 ±0.34 | 21.60 ±0.40 | 13.66 ±0.25 |
| Sieved | 24.92 ±0.25 | 26.69 ±0.32 | 23.31 ±0.25 |
| 2–5 inches† | | | |
| In place (top) | 22.12 ±0.57 | 20.01 ±0.64 | 13.62 ±0.18 |
| In place (1.5 inches) | 20.06 ±0.78 | 21.18 ±0.35 | 12.94 ±0.17 |
| Sieved | 18.44 ±0.65 | 23.80 ±0.31 | 13.91 ±0.09 |
| 10–13 inches† | | | |
| In place (top) | 14.02 ±1.17 | 32.24 ±1.41 | 22.70 ±0.71 |
| In place (1.5 inches) | 11.26 ±0.56 | 32.67 ±1.33 | 25.10 ±0.73 |
| Sieved | 11.83 ±0.83 | 34.32 ±1.31 | 28.61 ±1.39 |
| 23–27 inches† | | | |
| In place (top) | 14.11 ±0.81 | 38.00 ±1.36 | ..... |
| In place (1.5 inches) | 14.13 ±0.50 | 36.38 ±1.53 | ..... |
| Sieved | 14.17 ±0.77 | 37.97 ±1.24 | ..... |

* 20 determinations on each area for every mean.

† 9 determinations on each area for every mean.

Duplicate determinations of the organic matter in the soil horizons showed that the surface soils contain a higher percentage than other horizons and that the amount decreases with increasing depth. The presence of the larger amounts of organic matter in the surface soils may account for at least part of the increased moisture equivalents at the surface, since the amounts of colloids in the $A_2$ horizons are approximately the same as at the surface and therefore likely do not cause the differences.

Throughout the Georgeville and Orange soil profiles the values obtained from the undisturbed soil are slightly lower than the values from sieved soil. This agrees with results obtained by Davis and Adams (5), who found that blocks of undisturbed soil give slightly lower moisture equivalent values than do crushed samples of the same material. Very likely the following conditions

obtain in regard to the two soil profiles. The undisturbed soil is apparently structurally arranged so that small drainage passages exist and allow better drainage of the water during centrifuging than that of the sieved soil, which has had the passages broken up. The removal of gravel from the sieved soil also may have brought about an increase in values for the sieved soil.

The two sets of moisture equivalent values in the Congaree profile differ very little; the values obtained from the sieved soil of the upper horizons being only slightly lower than those from undisturbed soil. This is the reverse of the situation in the Orange and Georgeville soil profiles. There are apparently

TABLE 3

*Mean wilting percentages with standard errors, wilting coefficients, and ratio of moisture equivalent to wilting percentage in the Congaree, Georgeville, and Orange soils at the depths indicated*

| SAMPLE | CONGAREE | GEORGEVILLE | ORANGE |
|---|---|---|---|
| Surface (0–1.5 inches) | | | |
| Wilting percentage* | 7.68 ±0.23 | 6.16 ±0.31 | 4.31 ±0.21 |
| Wilting coefficient | 16.25 | 13.64 | 10.11 |
| Ratio† | 3.89 | 4.07 | 4.32 |
| 2–3.5 inches | | | |
| Wilting percentage‡ | 5.34 ±0.63 | 5.54 ±0.43 | 3.03 ±0.08 |
| Wilting coefficient | 11.68 | 10.76 | 7.01 |
| Ratio† | 4.03 | 3.57 | 4.26 |
| 10–11.5 inches | | | |
| Wilting percentage‡ | 2.24 ±0.33 | 20.51 ±1.12 | 10.41 ±0.51 |
| Wilting coefficient | 6.88 | 17.47 | 12.72 |
| Ratio† | 5.65 | 1.57 | 2.25 |
| 23–24.5 inches | | | |
| Wilting percentage‡ | 4.58 ±0.61 | 22.85 ±1.27 | ..... |
| Wilting coefficient | 7.58 | 20.54 | ..... |
| Ratio† | 3.05 | 1.65 | ..... |

* 20 determinations on each area for every mean.

† Ratio is moisture equivalent divided by wilting percentage.

‡ 9 determinations on each area for every mean.

few drainage passages as a result of structural arrangement of the particles in the Congaree soil, and therefore when the soil aggregates are partly broken up better drainage of water occurs through the soil, because the coarse texture of the Congaree soil allows a smaller amount of compaction on centrifuging than in the two fine-textured soils.

The results of the determinations of the wilting percentages are presented in table 3. The wilting percentages of all horizons follow the general trend of the moisture equivalent values. In the Congaree soil profile the $A_1$ horizon

has the highest wilting percentage. In horizon 3 the minimum value is found, and this increases gradually in horizons 4 and 5. The whole general trend throughout the profile is probably a result of the same factors that influence moisture equivalent values. This is probably true of the Georgeville and Orange profiles as well.

In order to have wilting coefficients for exactly the same depths as the average depth of the samples from which the wilting percentages were determined, moisture equivalent values were estimated from curves of moisture equivalent in place. The estimated values were used to calculate the wilting coefficients which are presented in table 3. The moisture equivalents were also divided by the wilting percentages, and the ratios are presented in the same table. The ratio of the moisture equivalent to the wilting percentage varies from 1.57 to 5.65 and therefore does not agree with the determinations of Briggs and Shantz (2) on other soils, where the ratio was found to be approximately 1.84. No reason for this wide difference can be given on the basis of the data obtained in this study. The data of Veihmeyer and Hendrickson (9) show that the residual moisture at permanent wilting cannot be obtained in every case from the moisture equivalent by use of the factor 1.84. They found ratios from 1.73 to 3.82. From the differences that exist between the wilting percentage and wilting coefficient values in the present study it is apparent that the wilting coefficient is not a good measure of the lower limit of available water in the three forest soils, Congaree clay loam, Georgeville clay, and Orange loam.

## SUMMARY

Soil analyses and determinations of moisture equivalent, wilting coefficient, and wilting percentage were made of three forest soils of the Duke Forest, Congaree clay loam, Georgeville clay, and Orange loam.

Each of the experimental areas is described briefly.

Experimental methods are described.

Moisture equivalent values were found to be higher in sieved than in unsieved soil in the Georgeville and Orange soils.

Moisture equivalent values for the lower soil horizons appear to be directly correlated with colloid content. Organic content appears to be directly correlated with the values for the surface horizons.

Wilting percentages, though much lower, were found to follow the general trend of the moisture equivalent values. The wilting percentage values are apparently conditioned by the same factors that influence the moisture equivalent values.

The ratio of moisture equivalent to wilting percentage was found to vary from 1.57 to 5.65.

Wilting coefficient values were found to vary considerably from those of the wilting percentage. Wilting coefficient, therefore, is not a good measure of the lower limit of available water in Congaree clay loam, Georgeville clay, and Orange loam soils of the Duke Forest.

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# THE INFLUENCE OF CROPPING PRACTICES ON SOME PHYSICAL AND CHEMICAL PROPERTIES OF SOIL[1]

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The soil conservation program for the Piedmont Plateau includes the use of close-growing crops, chiefly legumes such as Austrian winter peas, crimson clover, vetch, soybeans, and lespedeza, as an effective means for the control of sheet erosion on terraced lands. The protection afforded by these crops may be attributed to a combination of factors such as the interception of rainfall by the vegetative covering, the mechanical effect of plants and plant roots, and the alteration of the structure of the soil.

Data presented by Middleton, et al. (12, 13), Peele and Moser (17), and Diseker and Yoder (6) showed that soil and water losses from cultivable areas were materially reduced when legume crops were included in the rotation. Haynes[3] has shown that the maximum interception of rainfall by the plant canopy is reached as the plant approaches maturity. Lyon and Buckman (10, p. 48–52) point out that organic matter plays an important role in the formation of a desirable soil structure by increasing the porosity and absorptive capacity, by improving the tilth through the formation of a crumb structure, and by developing larger aggregates.

Until recently, only meager quantitative data were available on the relationship between physical properties of soil and its erodibility. Data reported by Musgrave (15), Baver (2), Browning (5), and Peele (16) showed that organic matter increased percolation and aided in formation of larger stable aggregates. Musgrave (15) suggested, however, that organic matter may not always increase the rate of water movement through soil, for in the Upshur soil organic matter reduced the rate of water movement. His hypothesis is that the retardation in this soil was due to the presence of active inorganic colloids. A decrease in percolation was also reported by Lyon et al. (9), in lysimeter experiments on Dunkirk silty clay loam. The tanks without legumes

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[2] Acknowledgment is made to Bryan De Young, S.C.S., for servicing erosion control plats and to O. W. Beal, S.C.S., for certain laboratory analyses, as dispersion ratio, suspension percentages, and mechanical analysis.

[3] Haynes, J. L. 1937 Interception of rainfall by vegetative canopy. Mimeograph. Cir. of Symposium of Vegetative Method of Erosion Control. Ann. Meeting Amer. Soc. Agron.

in the rotation gave higher percolation rates than did the tanks with legumes in the rotation.

### EROSION LOSSES MEASURED UNDER FIELD CONDITIONS

Differences in soil and water losses resulting from various cropping systems were obtained from erosion control plats on Cecil sandy clay loam at Tigerville, Duncan, and Greer, South Carolina. These experiments were designed

TABLE 1

*Monthly rainfall, run-off, and soil loss under different cropping systems designed for erosion control on Cecil sandy clay loam at Tigerville, S. C.*

| MONTH | RAINFALL | CROPPING SYSTEM | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Corn-cotton | | Corn-cotton-(vetch) | | Oats-(lespedeza)-cotton | | Fallow | |
| | | Run-off | Soil loss | Run-off | Soil loss | Run-off | Soil loss | Run-off | Soil loss |
| | *inches* | *per cent* | *tons/A.* | *per cent* | *tons/A.* | *per cent* | *tons/A.* | *per cent* | *tons/A.* |
| *1937* | | | | | | | | | |
| January | 11.67 | 32.59 | 0.60 | 19.42 | 0.09 | 0.67 | 0.00 | 30.67 | 1.21 |
| February | 3.95 | 8.59 | 0.24 | 5.98 | 0.04 | 0.27 | 0.00 | 5.12 | 0.20 |
| March | 2.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| April | 5.47 | 7.33 | 0.03 | 4.21 | 0.01 | 2.61 | 0.01 | 21.57 | 4.18 |
| May | 3.18 | 10.43 | 0.16 | 3.95 | 0.02 | 9.60 | 2.04 | 37.32 | 10.12 |
| June | 3.44 | 18.16 | 4.91 | 13.09 | 2.54 | 17.48 | 5.19 | 36.99 | 14.89 |
| July | 5.40 | 24.19 | 4.93 | 18.99 | 2.34 | 12.35 | 1.97 | 28.40 | 9.72 |
| August | 8.65 | 41.59 | 9.12 | 38.26 | 5.01 | 26.50 | 3.43 | 40.50 | 14.60 |
| September | 4.56 | 60.09 | 1.90 | 46.18 | 1.97 | 32.00 | 0.38 | 40.62 | 10.32 |
| October | 9.08 | 41.47 | 0.44 | 4.72 | 0.03 | 1.01 | 0.00 | 39.50 | 0.54 |
| November | 1.14 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| December | 2.35 | 0.02 | 0.00 | 0.00 | 0.00 | 0.04 | 0.00 | 0.12 | 0.00 |
| *1938* | | | | | | | | | |
| January | 3.24 | 0.39 | 0.00 | 0.03 | 0.00 | 0.12 | 0.00 | 0.78 | 0.01 |
| February | 1.96 | 0.05 | 0.00 | 0.00 | 0.00 | 0.10 | 0.00 | 0.10 | 0.00 |
| March | 6.07 | 8.12 | 0.11 | 0.48 | 0.00 | 7.16 | 0.14 | 25.20 | 6.78 |
| April | 2.25 | 1.59 | 0.00 | 0.08 | 0.00 | 1.34 | 0.00 | 6.01 | 0.11 |
| May | 4.40 | 0.19 | 0.00 | 0.19 | 0.00 | 8.60 | 0.05 | 24.59 | 5.27 |
| June | 3.49 | 0.39 | 0.00 | 0.36 | 0.00 | 7.05 | 0.12 | 10.45 | 3.50 |
| Total | 82.32 | 19.98 | 22.44 | 12.28 | 12.05 | 8.10 | 13.33 | 25.20 | 81.45 |

to collect the soil and water eroding from 1/100-acre plats. Suitable calibrated catchment tanks were placed at the bottom of each plat for this purpose. After each rain the amount of run-off was measured, and an aliquot of the eroded material was brought to the laboratory to determine the total soil loss from each rotation. Monthly summaries of rainfall and soil and water losses for the erosion control plats under various cropping systems are presented in tables 1, 2, and 3. The average run-off (table 4) shows significant differences for various cropping systems, but does not show the comparative amounts of

erosion occurring under the different rotations. The results from the Tigerville experiment show that erosion was markedly reduced during periods when the legumes afforded the maximum cover. In the oats-(lespedeza)-cotton rotation, the run-off and soil losses were highest during the early part of the first season when lespedeza was becoming established, but were almost nil during the latter part of the season and throughout the entire second season. The vetch cover crop had a similar retarding effect on erosion except that the

TABLE 2

*Monthly rainfall, run-off, and soil loss under different cropping systems designed for erosion control on Cecil sandy clay loam at Duncan, S. C.*

| MONTH | RAINFALL | CROPPING SYSTEM | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Corn-cotton | | Oats-(lespedeza)-cotton | | Oats-(soybeans)-cotton | | Fallow | |
| | | Run-off | Soil loss | Run-off | Soil loss | Run-off | Soil loss | Run-off | Soil loss |
| | *inches* | *per cent* | *tons/A.* | *per cent* | *tons/A.* | *per cent* | *tons/A.* | *per cent* | *tons/A.* |
| *1937* | | | | | | | | | |
| January | 10.73 | 34.94 | 0.22 | 34.32 | 0.55 | 8.54 | 0.02 | 36.76 | 0.58 |
| February | 3.59 | 5.20 | 0.01 | 6.18 | 0.08 | 0.60 | 0.00 | 8.53 | 0.09 |
| March | 2.25 | 0.42 | 0.00 | 0.24 | 0.00 | 0.00 | 0.00 | 0.20 | 0.00 |
| April | 5.65 | 4.80 | 0.03 | 13.67 | 0.11 | 8.51 | 0.08 | 15.96 | 1.33 |
| May | 3.71 | 2.66 | 0.02 | 25.83 | 0.22 | 26.23 | 0.58 | 33.76 | 0.52 |
| June | 6.50 | 42.87 | 18.44 | 54.53 | 0.28 | 45.70 | 21.55 | 61.88 | 19.01 |
| July | 2.88 | 27.52 | 0.86 | 19.11 | 0.15 | 15.53 | 2.30 | 29.61 | 6.55 |
| August | 4.33 | 24.09 | 0.39 | 9.55 | 0.01 | 17.39 | 0.30 | 22.06 | 1.20 |
| September | 3.92 | 49.09 | 0.57 | 31.70 | 0.00 | 43.57 | 0.33 | 43.33 | 4.35 |
| October | 10.66 | 46.49 | 0.88 | 16.47 | 0.00 | 32.12 | 0.26 | 42.28 | 1.26 |
| November | 1.29 | 0.84 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| December | 2.13 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| *1938* | | | | | | | | | |
| January | 2.55 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| February | 1.10 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| March | 3.27 | 2.51 | 0.01 | 0.12 | 0.00 | 0.18 | 0.00 | 5.99 | 0.05 |
| April | 2.02 | 0.34 | 0.00 | 0.06 | 0.00 | 0.06 | 0.00 | 5.46 | 0.01 |
| May | 2.89 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.28 | 0.00 |
| June | 2.47 | 18.31 | 0.36 | 0.66 | 0.00 | 15.60 | 0.58 | 32.86 | 2.28 |
| Total | 71.94 | 22.81 | 21.79 | 18.31 | 1.40 | 16.80 | 26.00 | 27.09 | 37.23 |

cultivation of the soil required in seeding the crop was partly responsible for the low erosion losses in September. After the crop was established, a marked reduction in erosion occurred, and soil and water losses were almost negligible during the period of maximum growth.

The effects of legumes on erosion control were not so pronounced at Duncan (table 2) as at Tigerville. The oats-(lespedeza)-cotton rotation was started after the outset of the experiment, and very low amounts of rainfall occurred during the effective growth period for lespedeza, which is normally the second

year. As a result, the run-off differences were not so pronounced as they would probably have been if a normal amount of precipitation had occurred during the first 6 months of 1938.

The effects of the legume at Duncan and Greer were also minimized by the initial high organic matter content of this soil, as lespedeza had been included in the crop rotation practiced on the field several years before the control plats were established. The legume plats in both of these experiments, however, gave a reduced soil and water loss in comparison with the non legume

TABLE 3

*Monthly rainfall, run-off, and soil loss under different cropping systems designed for erosion control on Cecil sandy clay loam at Greer, S. C.*

| MONTH | RAINFALL | CROPPING SYSTEM | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Corn-cotton | | Corn-cotton-(vetch) | | Oats-(lespedeza)-cotton | | Oats-(soybeans)-cotton | | Fallow | |
| | | Run-off | Soil loss | Run-off | Soil loss | Run-off | Soil loss | Run-off | Soil loss | Run-off | Soil loss |
| | *inches* | *per cent* | *tons/A.* | *per cent* | *tons/A.* | *per cent* | *tons/A.* | *per cent* | *tons/A.* | *per cent* | *tons/A.* |
| *1937* | | | | | | | | | | | |
| April | 6.15 | 3.32 | 0.06 | 2.81 | 0.03 | 13.22 | 1.18 | 13.63 | 1.56 | 25.89 | 2.10 |
| May | 3.59 | 5.91 | 0.04 | 3.31 | 0.01 | 27.79 | 0.13 | 28.40 | 0.22 | 39.52 | 0.38 |
| June | 6.55 | 48.83 | 2.59 | 47.47 | 2.32 | 49.50 | 0.72 | 52.45 | 0.93 | 66.15 | 6.96 |
| July | 2.71 | 13.14 | 0.19 | 9.12 | 0.02 | 17.02 | 0.13 | 8.08 | 0.35 | 27.61 | 3.06 |
| August | 4.94 | 22.15 | 0.07 | 13.32 | 0.06 | 18.87 | 0.13 | 17.87 | 0.12 | 40.80 | 0.57 |
| September | 4.50 | 41.94 | 0.12 | 49.14 | 0.16 | 45.33 | 0.07 | 48.40 | 0.22 | 56.90 | 3.84 |
| October | 10.91 | 29.97 | 0.08 | 21.08 | 0.15 | 23.26 | 0.02 | 20.21 | 0.06 | 57.85 | 2.67 |
| November | 1.47 | 0.86 | 0.00 | 0.53 | 0.00 | 0.92 | 0.00 | 0.59 | 0.00 | 0.79 | 0.00 |
| December | 2.26 | 1.00 | 0.00 | 1.06 | 0.00 | 0.81 | 0.00 | 0.77 | 0.00 | 0.26 | 0.00 |
| *1938* | | | | | | | | | | | |
| January | 2.82 | 1.74 | 0.00 | 2.43 | 0.00 | 1.32 | 0.00 | 1.24 | 0.00 | 1.31 | 0.00 |
| February | 1.19 | 0.89 | 0.00 | 0.48 | 0.00 | 0.77 | 0.00 | 0.65 | 0.00 | 0.08 | 0.00 |
| March | 3.59 | 2.45 | 0.00 | 1.38 | 0.00 | 1.60 | 0.00 | 2.28 | 0.02 | 13.19 | 0.40 |
| April | 2.23 | 0.26 | 0.00 | 0.22 | 0.00 | 1.48 | 0.00 | 0.80 | 0.00 | 2.09 | 0.00 |
| May | 3.44 | 0.37 | 0.00 | 0.22 | 0.00 | 1.39 | 0.00 | 0.28 | 0.00 | 9.31 | 0.10 |
| June | 2.47 | 3.85 | 0.02 | 5.86 | 0.02 | 1.85 | 0.00 | 5.67 | 0.04 | 20.17 | 0.18 |
| Total | 58.82 | 16.31 | 3.17 | 13.60 | 2.77 | 14.17 | 2.38 | 13.64 | 3.52 | 16.35 | 20.26 |

plat, but the differences were more pronounced during the period when the plats provided the maximum cover.

The oats-(soybeans)-cotton rotation system afforded some protection, but was not nearly so effective as the cropping practice including either lespedeza or vetch. The soil planted to the oats-(soybeans)-cotton rotation eroded more readily during the early growth stage of the soybeans than was the case in the the corn-cotton rotation, but a reduced erosion loss occurred during the late fall and winter months. Apparently some differences occurred in soil structure, as there was less erosion during the following season. Some of the

beneficial effects of the soybeans may have been due to the fissures in the soil formed by the taproots.

The data from these experiments show not only that the erosion losses were retarded during the actual growing periods of the legumes, but that the soil

TABLE 4

*Influence of various cropping systems on soil properties associated with water movement and average annual run-off*

| LOCATION | CROPPING SYSTEM | ORGANIC MATTER | HUMUS | PERCOLATION | RUN-OFF | AGGREGATE SIZES | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | 1.0 mm. | 1.0–0.5 mm. | 0.5–0.05 mm. | 0.05–0.02 mm. | <0.02 mm. |
| | | *per cent* | *per cent* | *in./hr.* | *per cent* | *per cent* | *per cent* | *per cent* | *per cent* | *per cent* |
| Tigerville | Before cropping* | 1.20 | 0.18 | 10.92 | ..... | 7.43 | 19.25 | 58.42 | 5.03 | 9.87 |
| | Corn-cotton | 1.18 | 0.17 | 5.13 | 19.85 | 8.10 | 17.20 | 58.00 | 7.30 | 9.40 |
| | Corn-cotton-(vetch) | 1.46 | 0.23 | 12.52 | 15.09 | 12.71 | 21.17 | 53.22 | 7.90 | 5.10 |
| | Oats-(lespedeza)-cotton | 1.48 | 0.23 | 7.94 | 6.58 | 10.02 | 19.55 | 56.93 | 7.05 | 7.45 |
| | Bermuda sod | 2.76 | 0.61 | 12.28 | 0.16 | 17.87 | 22.25 | 47.38 | 9.20 | 4.40 |
| | Fallow | 1.10 | 0.11 | 10.73 | 30.25 | 12.22 | 22.39 | 53.37 | 5.50 | 6.00 |
| Duncan | Before cropping* | 2.10 | 0.12 | 11.98 | ..... | 8.97 | 12.48 | 66.65 | 8.37 | 4.53 |
| | Corn-cotton | 2.09 | 0.14 | 12.27 | 27.02 | 18.50 | 17.25 | 52.56 | 6.91 | 4.77 |
| | Oats-(lespedeza)-cotton | 2.43 | 0.21 | 12.21 | 19.52 | 15.34 | 13.92 | 60.68 | 6.80 | 4.26 |
| | Oats-(soybeans)-cotton | 2.05 | 0.13 | 13.87 | 23.71 | 12.35 | 13.31 | 64.04 | 5.47 | 4.87 |
| | Bermuda sod | 2.45 | 0.45 | 12.18 | 0.42 | 20.10 | 18.72 | 51.88 | 4.74 | 4.65 |
| | Fallow | 1.56 | 0.12 | 10.83 | 31.22 | 10.46 | 12.17 | 64.53 | 8.18 | 4.78 |
| Greer | Before cropping* | 2.52 | 0.21 | 10.61 | ..... | 12.02 | 18.21 | 56.77 | 5.44 | 7.66 |
| | Corn-cotton | 2.53 | 0.22 | 12.18 | 16.31 | 19.00 | 16.01 | 50.99 | 5.44 | 8.87 |
| | Corn-cotton-(vetch) | 2.61 | 0.27 | 15.62 | 13.60 | 21.57 | 20.76 | 45.17 | 6.05 | 6.05 |
| | Oats-(lespedeza)-cotton | 2.70 | 0.27 | 12.82 | 14.17 | 17.01 | 18.99 | 52.36 | 6.37 | 6.37 |
| | Oats-(soybeans)-cotton | 2.57 | 0.24 | 10.86 | 13.64 | 21.42 | 19.31 | 47.25 | 5.35 | 6.47 |
| | Fallow | 2.52 | 0.21 | 12.18 | 30.64 | 16.35 | 14.75 | 53.94 | 8.19 | 6.87 |
| Clemson | Cotton | 1.23 | 0.11 | 13.42 | ..... | 7.00 | 27.72 | 48.90 | 7.16 | 9.22 |
| | Cotton (vetch) | 1.72 | 0.24 | 18.20 | ..... | 7.53 | 25.73 | 54.68 | 7.21 | 4.85 |
| | Cotton, 8 T. manure | 1.76 | 0.23 | 16.86 | ..... | 9.31 | 27.58 | 52.52 | 6.35 | 4.44 |
| | Cotton-(vetch), 8 T. manure | 1.87 | 0.22 | 17.27 | ..... | 6.20 | 32.65 | 49.63 | 7.03 | 4.62 |

* Data represent analyses of original soil.

was less susceptible to erosion during the succeeding year when cultivated crops were growing. These differences suggested that an investigation of certain physical properties of these soils might give some additional information on the soil structure which persists in the soil after the cover crop has been turned under as green manure.

## EXPERIMENTAL PROCEDURE

Soil samples were taken at the outset and at the end of the experiment from each of the erosion control plats, and also from certain plats at Clemson designed to study crop responses from additions of organic matter applied in the form of barnyard manure and from vetch as green manure. These samples were taken to the laboratory to determine the chemical and physical differences associated with erodibility of soils under field conditions.

The amount of organic carbon was determined by the dry combustion method (1, p. 4), and the results were converted into organic matter by multiplying by the factor 1.724; the humus content was obtained by extraction with a 2.5 per cent NaOH solution followed by precipitation with HC1 according to the method of Waksman (20). Analyses of the soil aggregates were made by a modification of the method outlined by Bouyoucos (3). After passing through the 0.5-mm. sieve, the soil suspension was transferred to a hydrometer jar. The readings with a Bouyoucos soil hydrometer were made at various time intervals corresponding to settling velocities of various sized particles as calculated by the Stokes formula. Mechanical analysis was determined by the pipette method of Olmstead, et al. (14), moisture equivalent by the method of Briggs and McLane (4), and saturation capacity by the method of Keen and Raczkowski (7).

Field volume weight, or apparent specific gravity, was determined by the procedure of King (8), field capacity moisture by the method inaugurated by Shaw (18), and percolation was determined in artificial packed tubes, being expressed in inches per hour according to the formula of Slater and Byers (19). The volume of macropores was calculated by computing the total pore space by the usual method and subtracting the volume occupied by the micropores, which was calculated by multiplying the moisture equivalent by the volume weight of the soil.

## SOIL PROPERTIES AS AFFECTED BY CROPS

A comparison of the organic matter, humus content, rate of percolation, and size of aggregates is shown in table 4 together with the run-off percentage under field conditions. The organic matter content at the outset of the experiment at Tigerville was 1.20 per cent, at Duncan 2.10 per cent, and at Greer 2.52 per cent. At Tigerville, appreciable changes have occurred during the experiment under the various cropping systems. Lespedeza and vetch were effective in increasing the organic matter content, while a gradual depletion of organic matter occurred in the fallow plat. At Duncan, the same general effects resulted from identical cropping systems. Bermuda grass had the most influence on increasing the content of organic matter, while soybeans maintained a level approximately at the initial amount. Since the soybeans were removed as hay no marked increase was expected. At Greer the effects of the legumes were somewhat minimized, probably because of the high initial

level of organic matter; however, the soil-conserving rotations showed slight increases when compared with either the fallow plat or the corn-cotton rotation.

The results from certain plats at Clemson were also included with these data in order to obtain the cumulative effects of 12 annual applications of stable manure, manure plus a vetch cover crop, and vetch turned under as green manure upon physical properties of the soil. The percentage of organic matter in the soil from the manure and vetch plats was higher than that in the soil from the continuous cotton plat.

The effects of the various crops on soil aggregation show some differences. The data from the Tigerville and Clemson plats are probably more significant than those from the other locations, as these experiments had been in progress for a longer time. The differences in percentages of various sized aggregates are more evident in the <0.02-mm. size than in the other divisions. At Tigerville, a lower percentage of small aggregates, with the exception of the fallow soil, and a higher percentage of 0.05–0.02-mm. particles were found in the soil from the Bermuda grass sod than in the other soils. Lespedeza and vetch were not quite so effective in the formation of soil aggregates as was the Bermuda grass, but a lower percentage of particles <0.02 mm. was found in the soil from the rotations including these legumes than from the nonlegume rotation. The effects of crops on aggregate formation were somewhat nullified in the Duncan and Greer experiments by the higher organic matter levels of these two soils. As a consequence, the percentage of particles <0.02 mm. was almost identical on all cropped plats. Metzger and Hyde (11) in their recent work suggested that organic matter encourages soil aggregation.

The fallow plat at Tigerville showed a lower percentage of aggregates in the 0.05–0.02-mm. range than did any of the cropped soils, a result which was contrary to the relation between organic matter and soil aggregation. Erosion in this case had removed the major portion of the surface soil, and the upper $6\frac{2}{3}$ inches of soil contained a considerable amount of the B horizon. Mechanical analysis of the soil showed that the clay content was higher in the fallow plat at the end than at the outset of the experiment. As subsurface soils usually are more highly aggregated than surface soils, a lower percentage of aggregates <0.02 mm. would be anticipated in the fallow plat.

Some irregularities appeared in the percolation rate. This may be influenced by numerous factors, among which the nature and extent of the organic matter are extremely important. The humus content was determined to obtain some idea of the relative amounts of the finely divided organic colloidal material, since the determination of total organic matter does not show the chemical nature of the material. The variation in content of humus was not so great as that found in total organic matter, which appeared to be associated with the type of plant material applied to the soil. Generally the run-off was high and the percolation rate was low where the humus content was low, whereas the run-off was lower and the percolation rate higher where the humus content was high. Apparently, percolation rates as determined in this experiment

TABLE 5

*Influence of various cropping systems on certain physical properties associated with erodibility of soils*

| LOCATION | CROPPING SYSTEM | VOLUME WEIGHT | WATER AT SATURATION | FIELD MOISTURE CAPACITY | MOISTURE EQUIVALENT | SUSPENSION PERCENTAGE (0.02 MM.) | VOLUME OF MACROPORES | DISPERSION RATIO (0.02 MM.) | MECHANICAL ANALYSIS | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | Sand | Silt | Clay |
| | | | *per cent* | | | | *per cent* | | *per cent* | *per cent* | *per cent* |
| Tigerville | Before cropping* | 1.19 | 35.36 | ..... | 15.89 | 5.27 | 36.19 | 14.69 | 65.91 | 11.82 | 22.27 |
| | Corn-cotton | 1.20 | 34.41 | 15.51 | 15.26 | 7.65 | 36.42 | 22.08 | 65.35 | 12.09 | 22.56 |
| | Corn-cotton-(vetch) | 1.16 | 36.48 | 17.61 | 15.45 | 5.32 | 38.45 | 15.63 | 65.91 | 11.55 | 22.54 |
| | Oats-(lespedeza)-cotton | 1.13 | 40.70 | 17.22 | 16.08 | 5.93 | 39.19 | 15.98 | 62.90 | 13.24 | 23.86 |
| | Bermuda sod | 1.12 | 37.37 | 20.42 | 17.70 | 5.65 | 36.81 | 15.79 | 64.25 | 14.58 | 21.17 |
| | Fallow | 1.26 | 32.47 | 15.36 | 16.11 | 6.04 | 23.24 | 15.94 | 62.11 | 13.83 | 24.06 |
| Duncan | Before cropping* | 1.25 | 45.21 | ..... | 15.70 | 3.59 | 33.22 | 11.19 | 70.56 | 10.48 | 19.94 |
| | Corn-cotton | 1.22 | 47.38 | 12.28 | 15.20 | 5.77 | 35.74 | 18.96 | 69.58 | 8.93 | 21.49 |
| | Oats-(lespedeza)-cotton | 1.16 | 47.74 | 15.65 | 16.14 | 4.84 | 38.08 | 16.56 | 70.64 | 8.23 | 21.13 |
| | Oats-(soybeans)-cotton | 1.26 | 45.65 | 13.08 | 15.47 | 5.04 | 32.97 | 16.54 | 69.54 | 11.29 | 19.17 |
| | Bermuda sod | 1.17 | 48.83 | 15.78 | 15.20 | 4.22 | 37.87 | 17.40 | 75.75 | 10.43 | 13.82 |
| | Fallow | 1.35 | 44.56 | 12.05 | 14.38 | 5.85 | 29.67 | 24.90 | 76.51 | 9.88 | 13.61 |
| Greer | Before cropping* | 1.24 | 35.47 | ..... | 14.30 | 4.40 | 35.48 | 13.99 | 71.73 | 10.75 | 17.52 |
| | Corn-cotton | 1.25 | 31.58 | 12.12 | 12.61 | 4.81 | 37.07 | 21.06 | 77.16 | 11.02 | 11.82 |
| | Corn-cotton-(vetch) | 1.24 | 32.57 | 13.37 | 12.57 | 3.11 | 37.62 | 14.45 | 78.47 | 11.27 | 10.26 |
| | Oats-(lespedeza)-cotton | 1.20 | 38.03 | 17.70 | 15.01 | 5.33 | 36.71 | 15.56 | 65.74 | 12.01 | 22.24 |
| | Oats-(soybeans)-cotton | 1.19 | 35.85 | 13.71 | 15.10 | 5.64 | 37.13 | 20.00 | 71.81 | 11.27 | 16.92 |
| | Fallow | 1.22 | 36.84 | 15.21 | 14.57 | 5.24 | 36.19 | 17.60 | 70.23 | 11.39 | 18.38 |
| Clemson | Cotton | 1.37 | 31.12 | 12.60 | 12.62 | 6.24 | 31.01 | 25.06 | 75.10 | 10.16 | 14.74 |
| | Cotton-(vetch) | 1.33 | 34.24 | 12.90 | 10.82 | 3.11 | 35.42 | 17.61 | 81.76 | 9.12 | 9.12 |
| | Cotton, 8 T. manure | 1.31 | 33.67 | 13.60 | 11.30 | 3.98 | 35.77 | 20.55 | 80.64 | 8.78 | 10.58 |
| | Cotton-(vetch), 8 T. manure | 1.32 | 34.28 | 13.01 | 11.05 | 4.83 | 35.60 | 24.07 | 79.95 | 8.82 | 11.23 |

* Date represent analyses of original soil.

cannot be directly correlated with run-off under field conditions, as the run-off may be influenced not only by soil conditions, but also by growing crops.

Other soil properties that are undoubtedly associated with erodibility of

soils are shown in table 5. The volume weight, saturation capacity, field moisture capacity, and moisture equivalent appeared to be directly related to one another and to the organic matter content. In all cases, volume weight definitely decreased when amounts of organic matter increased. There was not so much variation in the volume weight of the soil from the Greer experiment as from the other experiments, but this was attributed to the higher initial organic matter content of this soil. Bermuda grass sod produced the greatest effect on volume weight. The soil from the sod plat was capable of absorbing and retaining a higher percentage of moisture, as was shown by the water at saturation and the field moisture capacity, whereas the fallow plat had a higher volume weight and a relatively lower saturation capacity.

The percentage volume of macropores as shown for the various cropping systems indicated that soils well supplied with organic matter had the greatest effective porosity. Baver (2) suggested that soil porosity plays an important role in water movement and that the micropores are important largely in storage. Erosion, under the field conditions represented in this experiment, appeared to be associated with the volume of the macropores, as all the soil-conserving rotations had a greater effective porosity and produced a marked retardation in soil and water losses in comparison with the soil-depleting rotations, which had a lower percentage of macropores and larger soil and water losses. The continuous sod plat did not show such a direct correlation. The plat had a higher percentage of macropores but also had an extremely low erosion loss. The erosion losses from soil on these plats, however, were undoubtedly complicated by the vegetative effects of sod.

Either the suspension percentage or the dispersion ratio as shown for soils under various cropping systems appears to be a fair single criterion for prediction of erosional behavior of soils. The general correlations for these two evaluations of erodibility are that low suspension percentages and low dispersion ratios are prevalent in soils which have had legume crops included in the rotation systems; whereas higher suspension percentages and dispersion ratios are found in soils after nonlegume rotations. The effects of additions of organic matter to a soil are very conclusively demonstrated by these data, and undoubtedly organic matter, either directly or indirectly, has an important influence on the ultimate structure of the soil.

## SUMMARY

Studies on the effects of various cropping systems on the erodibility of soils are presented for plats at Tigerville, Duncan, and Greer. The data show that certain soil-conserving legume crops in a rotation not only reduced soil and water losses during the actual growing period of the legumes, but also made the soil less susceptible to erosion during the succeeding year when cultivated crops were grown.

A comparison of certain physical and chemical properties such as organic matter, humus, aggregation, volume weight, saturation capacity, field moisture

capacity, suspension percentage, dispersion ratio, percolation, and porosity were determined to note any correlation existing between erosion and soil properties affected by various cropping systems. The determinations of these soil properties under various cropping systems show considerable variations between soil-conserving rotations and soil-depleting rotations.

Organic matter content appears to be either directly or indirectly associated with the physical properties that are important in soil structure. Increases in the content of organic matter since the outset of the experiment resulted from rotations which included leguminous green manure crops, and decreases occurred in corn-cotton rotations, whereas the changes resulting from cropping systems were insignificant where the content of organic matter was abnormally high at the outset of the experiment.

Soil aggregation was influenced by the various erosion control crops. Bermuda grass sod increased the organic matter content, and as a result the soil under this type of cropping was in a high state of aggregation. The differences were most marked in the small sized aggregates, as indicated by the low percentage of $<0.02$ mm. aggregates, whereas a slight increase was noted in the $>0.5$ mm. aggregates. Vetch and lespedeza used in their respective rotations were not so effective as the Bermuda grass sod in promoting aggregation; however, a lower percentage of small aggregates was found on the lespedeza and vetch plats than on the corn-cotton rotations which did not include a legume.

Soils to which either green manure or barnyard manure was applied had lower volume weights, higher saturation capacities, and higher field moisture capacity than soils from soil-depleting rotations where only crop residues were added. This fact indicates a close correlation between these physical properties and organic matter.

Cropping practices also had an influence upon the dispersion ratio, suspension percentage, and porosity. Soil porosity, as indicated by the percentage volume of macropores, also appeared to be associated with erosion losses, as large soil and water losses usually occurred on soils with the lowest porosity, and smaller losses occurred on soils with higher porosity. The suspension percentage and the dispersion ratio showed significant correlations with erosional behavior of soils under field conditions and appeared to be good single indexes for predicting soil and water losses by laboratory methods.

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# EFFECT OF CHLOROPICRIN FUMIGATION ON NITRIFICATION AND AMMONIFICATION IN SOIL[1]

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Because of the interest that has developed in the use of volatile chemicals for partial soil sterilization The Rhode Island Agricultural Experiment Station has studied the use of chloropicrin ($CCl_3NO_2$). The primary objective in most experiments involving the use of this chemical for soil fumigation has been to control some specific, soil-harbored plant pathogene, little or no consideration being given to the effect which the treatment would have on the soil microorganism population as a whole. Where pathogenic organisms were controlled, crop yields were greatly increased, as might be expected. In some experiments where disease was felt to be of minor importance, however, large increases in growth and yields were obtained. For example, yields of carrots and onions were increased about 50 per cent, and pepper, tomato, and eggplant yields were at least doubled. In many such instances, because of the lack of apparent disease, it was difficult to explain entirely these unexpected increased yields on the basis of controlling any specific pathogenic organism. This work was initiated, therefore, to determine what effect the soil treatment has upon the summation of the metabolic activities of the flora and fauna of the soil.

## REVIEW OF THE LITERATURE

The relevant literature indicates that during the early part of the present century much work was done concerning the effect of so-called volatile antiseptics upon soil organisms and the effect of treatment on subsequent plant growth. It has been recognized that partial sterilization or treatment of the soil with heat or chemicals generally results in an increase in productivity of the soil. The vast amount of work done on this subject, however, has resulted in several conflicting statements and hypotheses. This confusion has undoubtedly arisen because of the fact that so many different factors are concerned with increasing plant growth. Variations would occur, for example, depending upon the agent used to sterilize the soil and upon the initial physical, chemical, and biotic condition of the soil treated.

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An excellent summary of early work on partial soil sterilization is presented by Kopeloff and Coleman (11). From this and more recent literature, it appears that the majority of the workers have ascribed the beneficial effects of partial sterilization to increased amounts of plant food, especially nitrogen, made available for plant use as a result of increased bacterial activity. Such workers as Russell and Hutchinson (13, 14), Hiltner and Störmer (9), Coleman (2), and Greig-Smith (6) seem to agree that increased bacterial activity is responsible, even though they may disagree as to just how this is brought about.

More recently Waksman and Starkey (18) have confirmed by experiment that partial sterilization results in an increase of the bacterial population and in ammonium-nitrogen accumulation. They explain this change as being due to a combination of factors; namely, changes in physical and chemical conditions of the soil, destruction of soil microorganisms thereby making their cell constituents available as a source of energy, changing the equilibrium of the microbiological flora, and the economical conversion of organic nitrogen to ammonium nitrogen.

Evidence has been reported in apparent opposition to the preceding concepts. Sirker (15) found that an equivalent amount of growth could not be obtained merely by the addition of large amounts of fertilizer. Gainey (4) demonstrated that increased yields could be obtained by treating the soil even though the numbers of bacteria were not increased. Also, Matthews (12) showed that chloropicrin treatment did not result in increasing the numbers of bacteria so much as did other antiseptics. Smith (16) in more recent work, however, has indicated that bacteria increased more rapidly in treated than in untreated soils.

Another explanation has been presented by Bolley (1) and others who believe that elimination of pathogenic fungi may have more to do with increase in crop growth than is ordinarily supposed. Jamieson (10) believes that the benefit is due to riddance in varying degrees of the forms of animal life in the soil that prey on plants. It has been generally recognized that the more unfavorable the initial biotic condition of the soil to plant development, the greater is the increase in subsequent productivity following treatment. Moreover, virtually all the workers have reported that an increase in yield could be expected wherever disease organisms were controlled by treatment.

Undoubtedly, the factors indicated by these workers were of major importance with regard to the particular soils or chemicals investigated. The status of chloropicrin, therefore, remains to be determined with regard to the effect which it has on the metabolism of the soil flora and fauna, the object being to determine whether there is an increase in bacterial activity which is of sufficient magnitude to increase the available nitrogen enough to account for some of the increases in growth recorded. Failure to find increasing bacterial activity would indicate the necessity of further investigations to ascertain whether or not beneficial effects are due to the destruction of some of the less evidently harmful organisms.

## EXPERIMENTATION

In order to measure the comparative nitrogen conversion activities of the soil organisms in treated and untreated soil, a modification of the method recommended by Waksman (17) for determining the rate of ammonification and nitrification was used. It consisted essentially of determining at intervals the rate of nitrification of ammonium sulfate and the ammonification and nitrification of dried blood. Thus, the effect of fumigating the soil with various dosages of chloropicrin on the subsequent activities of the surviving microorganisms during the growing season could be ascertained.

The soil used was Bridgehampton very fine sandy loam, taken from a field which had been cultivated for a number of years. This field had been fertilized with stable manure at the rate of 16 tons per acre the previous season, and it contained about 6.59 per cent organic matter. The H-ion concentration of a

TABLE 1

*Dosages of chloropicrin used for fumigation and the forms and amounts of nitrogen added*

| TREATMENT | DOSAGE OF CHLOROPICRIN PER CUBIC FOOT | NITROGEN COMPOUND ADDED | AMOUNT OF NITROGEN ADDED |
|---|---|---|---|
| | *cc.* | | *p.p.m.* |
| 1 | 0 | None | 0 |
| 2 | 0 | $(NH_4)_2SO_4$ | 300 |
| 3 | 1 | $(NH_4)_2SO_4$ | 300 |
| 4 | 3 | $(NH_4)_2SO_4$ | 300 |
| 5 | 9 | $(NH_4)_2SO_4$ | 300 |
| 6 | 0 | Dried blood | 265 |
| 7 | 1 | Dried blood | 265 |
| 8 | 3 | Dried blood | 265 |
| 9 | 9 | Dried blood | 265 |

composite sample was pH 6.15. It had a water-holding capacity of 54 per cent on the dry soil basis.

Partial sterilization was carried out in glazed earthenware crocks. The jars were nearly filled with 18 pounds of screened soil. Pure chloropicrin was applied in a single hole that extended almost to the bottom of the vessel. The amounts of chloropicrin applied were equivalent to dosages of 1 cc., 3 cc., and 9 cc. per cubic foot of soil. At the time of treatment, the soil contained approximately 21 per cent moisture on a dry weight basis. Immediately after treatment the jars were sealed with glue-coated paper. Four days after treatment the soil was removed from the crocks and spread out to aerate.

Twelve days after treatment samples from each of the various treatments and from the untreated soil were placed in pint widemouthed glass jars. Each sample contained the equivalent of 100 gm. of dry soil. After the samples were placed in the jars, ammonium sulfate was added to some, and dried blood to others, making nine different treatments of 32 samples each (table 1).

To those jars receiving ammonium sulfate, 210 mgm. of calcium carbonate was added to neutralize the resultant acidity.

In order to establish the nitrogen level at the time of the addition of the fertilizer, samples of soil to which no nitrogen compounds had been added, but which had received the various chloropicrin treatments, were analyzed for nitrates, nitrites, and ammonium nitrogen. This also indicated the chemical changes that had taken place during the 12-day aeration period. It was found that the 1-cc. dosage had no effect on nitrification, but that the 3-cc. and 9-cc. treatments had inhibited nitrification during this period as compared with the untreated soil. The ammonium-nitrogen content was about the same in all cases.

After the addition of the fertilizer the water content of each of the soil samples was raised to approximately 60 per cent of its water-holding capacity. The uncovered jars were placed in an oven and incubated at 28°C. A fan was placed in the incubator to ensure circulation of air. The moisture content of the soil was maintained at 50 to 60 per cent of its water-holding capacity by regular replenishment of the water lost.

Analyses were made at intervals by removing four jars from each of the nine treatments. Half of these were analyzed for nitrates and nitrites, and the other half for ammonium nitrogen. In this way duplicate results were obtained for each treatment. The nitrates were determined by the phenol-disulfonic acid method (7), the nitrites by the sulfanilic acid-naphthylamine method (3), and the ammonium nitrogen by Harper's method (8). Since changes took place more rapidly in the earlier part of the experiment, the intervals between the determinations were shorter during that period.

## RESULTS

The results obtained can best be presented in graphic form. The figures used in constructing the graphs were obtained by averaging results from duplicate samples.

In figure 1 note should be made that the untreated soil and the soil receiving 1 cc. chloropicrin per cubic foot had a nitrate-nitrogen content at the time of addition of the nitrogen compound of about 38 p.p.m.; whereas the soil treated with 3 cc. and 9 cc. contained about 18 p.p.m. of nitrate nitrogen. This was due to the fact that nitrification had taken place in the former case, from the time of treatment 12 days previous. In the soils receiving 3 cc. and 9 cc. of chloropicrin, however, nitrification had been inhibited during the period of aeration.

Further examination of figure 1 reveals that at the end of the 12-day period, nitrification was resumed in the 3-cc. treated soil. Moreover, the rate of nitrification was about parallel to that of the untreated and the 1-cc. treated soil. In the soil receiving 9 cc. chloropicrin, nitrification was inhibited for about 90 to 100 days. At that time either an unkilled residue of bacteria became active or the soil was reinoculated. Since retardation of nitrification

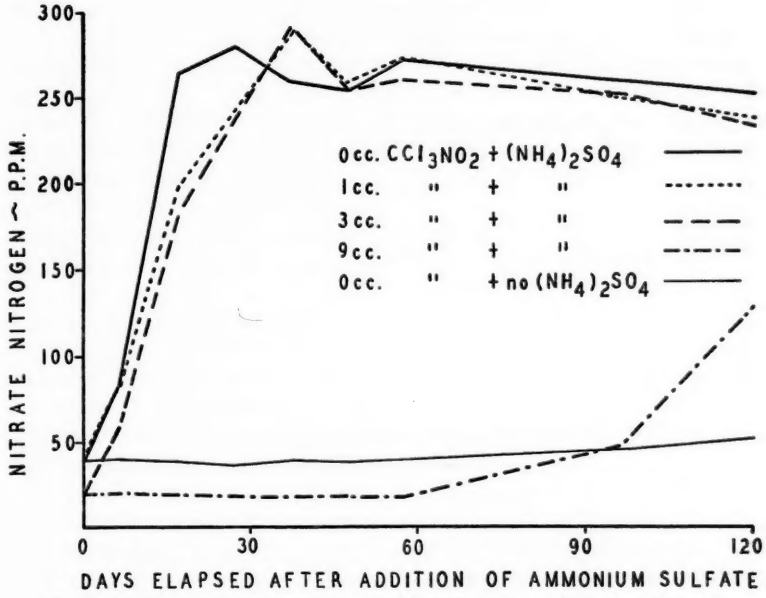


FIG. 1. EFFECT OF CHLOROPICRIN ON THE NITRIFICATION OF AMMONIUM SULFATE

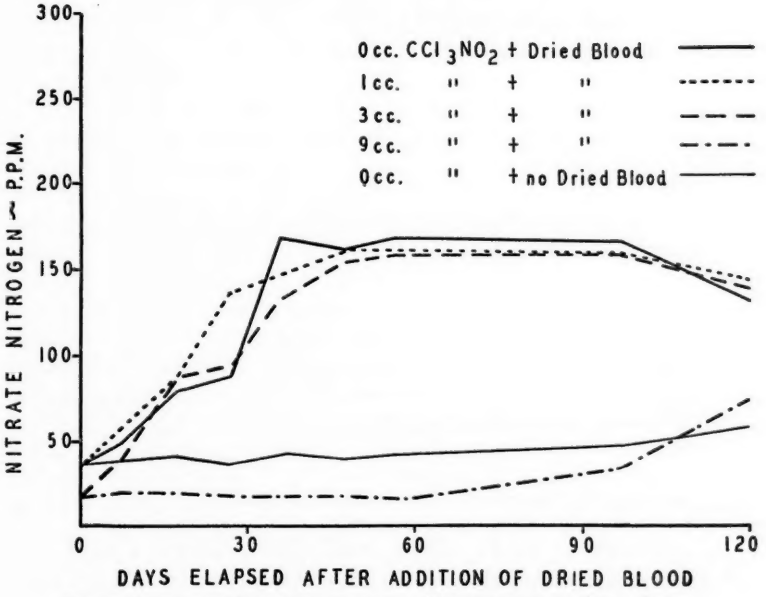


FIG. 2. EFFECT OF CHLOROPICRIN ON THE NITRIFICATION OF DRIED BLOOD

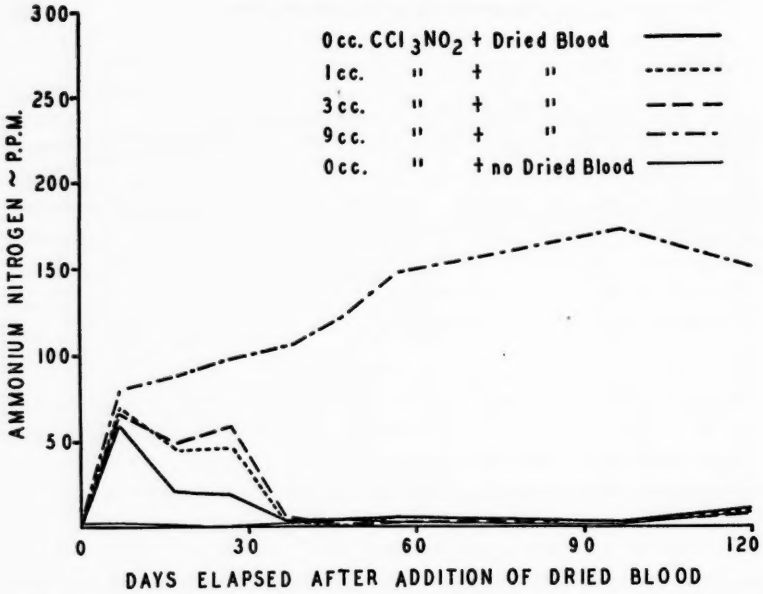


FIG. 3. EFFECT OF CHLOROPICRIN ON AMMONIUM-NITROGEN ACCUMULATION FROM DRIED BLOOD

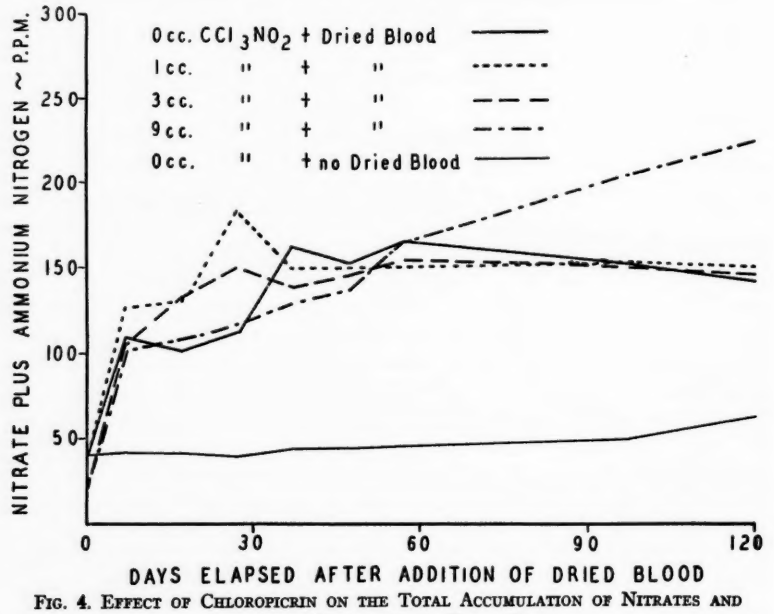


FIG. 4. EFFECT OF CHLOROPICRIN ON THE TOTAL ACCUMULATION OF NITRATES AND AMMONIUM NITROGEN FROM DRIED BLOOD

for a similar length of time was also observed in duplicate samples of the soils receiving the dried blood, it does not seem probable that nitrification was resumed because the soil had become reinfested. The rate of nitrification was slow even after the 90-day period. The resulting amount of nitrates, as well as the rate of nitrification in the 1-cc. and 3-cc. treated soils, was approximately the same as in the untreated soil. As ammonium-nitrogen determination on similarly treated soils revealed identical results, the graph showing the residual ammonium nitrogen has been omitted.

Figure 2 reveals that the amount of nitrates oxidized from the dried blood was about the same in the untreated and the 1-cc. and 3-cc. treated soils. In the soil receiving the 9-cc. treatment, nitrification was again inhibited. In figure 3 it may be seen that the 9-cc. treatment did not inhibit ammonification, but appears to have stimulated it slightly. Since this treatment inhibited nitrification, however, this graph shows an accumulation of ammonium nitrogen. In the untreated and in the 1-cc. and 3-cc. treated soils where nitrification was not seriously inhibited, ammonium nitrogen did not accumulate because it was immediately converted into nitrates.

In order to ascertain whether or not any of the treatments increased the total amount of nitrogen made available for plant use, the ammonium and nitrate nitrogen produced from the dried blood were added together. The results, as shown in figure 4, indicate that the soils receiving the 1-cc. and 3-cc. treatments produced no more available nitrogen than did the untreated soil. In the 9-cc. treated soil, however, there was a material increase in available nitrogen after 60 days. This increase produced by a high dosage is probably due to increasing the ammonifying power of the soil, but the effects appeared late in the experiment.

The nitrite-nitrogen determinations revealed that in no case did nitrites accumulate. The greatest amounts were found during the period when large amounts of ammonium nitrogen were being converted to nitrates. Even then, however, the nitrites amounted to only 2 to 3 p.p.m. Other than at this period, the nitrite-nitrogen content was negligible, being less than 1 p.p.m.

## DISCUSSION

The results indicate that in many respects chloropicrin behaves similarly to other so-called volatile antiseptics which have been tried for soil fumigation. Inhibition of nitrification has been reported by many workers who have used such substances as carbon disulfide and toluene. Chloropicrin is no exception to this, and it appears that increasing the dosage tends to increase the interval of time before active nitrification is resumed. Ammonification is not retarded, probably because many of the ammonifying bacteria are spore-formers and are thus difficult to kill. When nitrification is inhibited, ammonium nitrogen accumulates. Smith (16) in recent work confirms these results by finding that chloropicrin killed the nitrifying bacteria readily, whereas it had little effect upon the spore-forming bacteria. He obtained retardation of nitrification at

lower dosages of chloropicrin than are reported here, probably because the treatment was performed under more nearly optimum conditions.

Care should be taken not to assume that the results obtained under laboratory conditions will necessarily be duplicated under field conditions. The exact relationship is difficult to ascertain. In the laboratory the soil conditions can be easily controlled, and, more important, the fumigant can be confined for longer periods. The field dosage that resulted in increased yields was 2 cc. placed in holes 15 inches apart; less than the 3-cc. treatment used in this experiment. From other comparative tests with specific organisms under laboratory and field conditions, it does not seem probable that the field dosage mentioned would be sufficient to inhibit nitrification for any great length of time. Assuming that there was a retardation of nitrification in the field, the question arises as to what effect the resulting ammonium-nitrogen accumulation would have on plant growth. It does not seem probable, however, that the advantages of having nitrogen in the form of ammonium nitrogen instead of as nitrates could be of sufficient value to account for the increase in crop yields.

The outstanding difference between these results with chloropicrin and those obtained by investigators working with other chemicals, is the fact that neither the rate of nitrification nor the total amount of nitrogen made available was markedly increased over that in untreated soil. It is difficult to detect increased activity in treated over untreated soils when the rate of nitrification in the untreated soil is so rapid. The soil used in this experiment, however, was essentially the same as that which had responded well to chloropicrin fumigation, as reflected by increased crop yields. Because of the results obtained in this experiment, therefore, it does not seem probable that the increase in available nitrogen was responsible for the increase in yields obtained.

Assuming that increase in available nitrogen is not always the deciding factor in increasing crop yields, it seems advisable to turn toward another and more feasible explanation. It should be recalled, first, that the soils fumigated had been cropped for a number of years; second, that in plats where the greatest increases in yields were obtained, the same crop had been grown the previous year. Most workers, as indicated in the review of the literature, have reported that wherever disease organisms are controlled, increase in yields may be expected. Further, it has often been suggested that in virtually all soils that have been in use for a number of years, particularly for growing the same crop, there is an accumulation of undesirable organisms. The exact injury done to plant roots by these organisms, particularly fungi, is difficult to ascertain and is not generally given sufficient prominence. Since Smith (16), Godfrey (5), and others have shown chloropicrin to be a powerful lethal agent, particularly against fungi, it seems likely that the benefits of chloropicrin soil fumigation are in a large part due to the control of undesirable organisms, especially fungi.

It is probable, therefore, that plants grown in fumigated soil develop extensive, fibrous, uninjured root systems because they are subjected to fewer inhibitory organisms. This is reflected in vigorous growth and increased yield.

## SUMMARY

The effect of fumigating soil with chloropicrin upon the subsequent rate of ammonification and nitrification was studied.

Low dosages of chloropicrin were found to have little effect on nitrate formation; but, as the dosage was increased, nitrification was inhibited. The length of the inhibition period was dependent on the dosage of chloropicrin.

In no case was ammonification inhibited; therefore, in some instances the inhibition of nitrification resulted in an accumulation of ammonium nitrogen.

The total amount of nitrogen made available for plant growth was not materially increased except where high dosages of chloropicrin were used.

In view of the data obtained from this experiment the increases in plant growth obtained by treating the soil with low dosages of chloropicrin cannot be accounted for solely by the hypothesis that more nitrogen was made available for plant growth.

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